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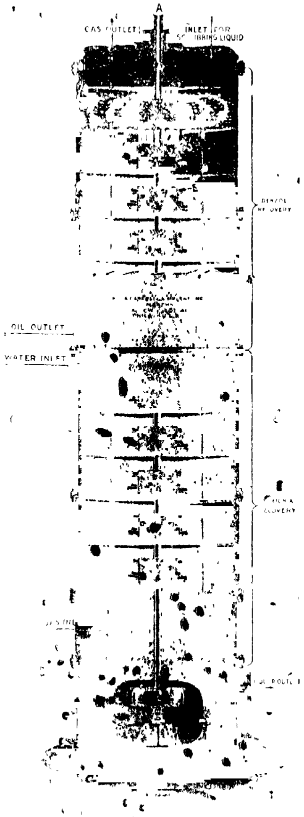

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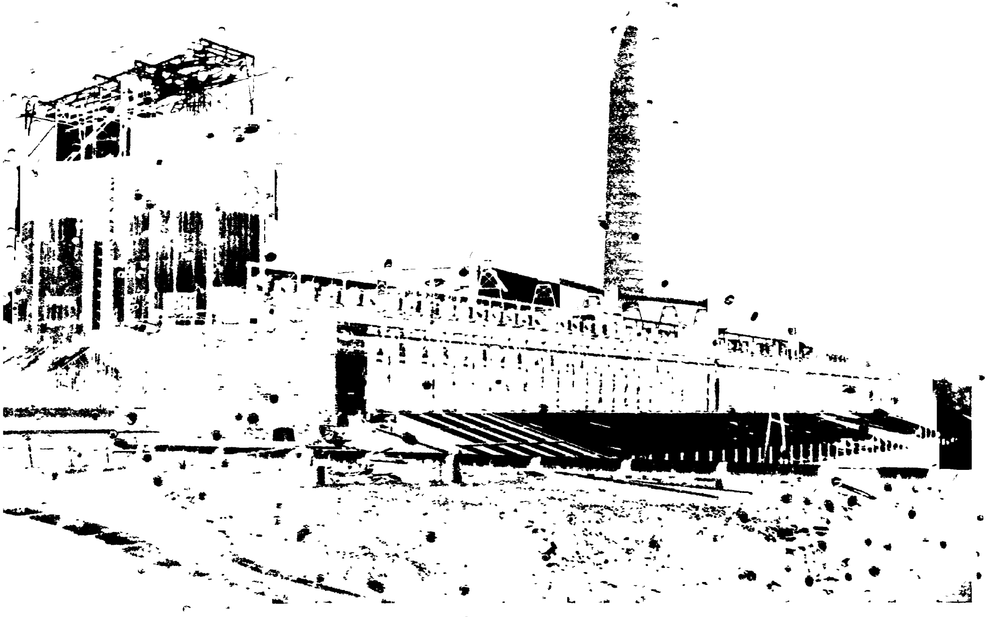

MODERN
COKING PRACTICE

J. E. CHRISTOPHER

RISEHOW COLLIERY COKING AND BY-PRODUCT CO.

Member of the Society of Chemical Industry, Late Lecturer on Fuels, etc., Wigan Technical College, Author of "Coal Distillation, Gasification, and By-Products"

INCLUDING THE

Analysis of Materials and Products

BY

T. H. BYROM

Fellow of the Institute of Chemistry, Fellow of the Chemical Society; Member of the Society of Chemical Industry, Late Chief Chemist to the Wigan Coal and Iron Co., for Fifteen Years Lecturer at the Wigan Technical College, Author of the "Physics and Chemistry of Mining"

Third Edition, Enlarged, in Two Volumes

Volume

RAW MATERIALS AND COKE

With numerous Illustrations and Folding Plates

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CROSBY LOCKWOOD AND SON
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PREFACE

SINCE the publication of the first edition in 1910, rapid progress has been made in Coking Practice, and the authors have realised for some considerable time the necessity for a second edition to bring the matter up to date. The publication has, however, been delayed through the exigencies of the European War, and in addition the authors have been unable to devote their time to the extent merited by the importance of the subject. It has been felt, however, that in view of the national aspect of the coking and by-product industry, the authors' continued experience should be embodied as soon as possible, and, in consequence, it has been thought advisable to issue the new edition in two volumes. The first volume dealing with Coking Practice, Raw Materials, etc., appears in its present form, to be followed by the second volume dealing with By-Products. Statistical matter has been brought up to date as far as possible, avoiding figures abnormally affected by the war conditions. The various chapters of the first edition have been considerably amplified, additional methods of analysis have been inserted, and certain physical tests have been introduced. The illustrations have for the most part been re-drawn. In general the coke ovens and appliances described are such as have found favour in the United Kingdom, and the authors are indebted to the various coke oven building firms for recent information. The authors have endeavoured as far as possible to quote references where matter outside their own experience has been inserted, and strongly recommend a perusal of the original matter so quoted. They conclude with the hope that the second edition of this work may play its part in bringing before the public the value of the coke and by-product industries as highly essential branches in the mining, metallurgical, and chemical industries of this country.

J. E. CHRISTOPHER.
T. H. BYROM.

May 1917

PREFACE TO THIRD EDITION

SINCE the publication of the second edition, the conclusion of the European War has left an aftermath of unsettled conditions and industrial unrest in this country which must seriously retard the development of the coking industry. Whilst endeavouring in this edition to bring statistical matter up to date, the authors consider it advisable to publish details of the most recent designs and methods in the form of appendices to each volume, in the hope that the time is not far distant when more stabilised conditions will allow the scientific and economic development of an industry which, during the recent years of national stress, has proved its value as a section of the key industry of coal carbonisation.

J. E. CHRISTOPHER.
T. H. BYROM.

September 1920

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MODERN COKING PRACTICE.

CHAPTER I

INTRODUCTION

COKE is the solid residue resulting from the destructive distillation or partial combustion of coal. The volatile matter is expelled by heat, leaving a residue, the character of which varies according to the nature of the coal treated, and according to the temperature to which the coal has been subjected.

The object of this "carbonisation" of coal is to obtain from it certain products, solid, liquid or gaseous, each having definite advantages over the raw coal in the industries in which they are to be employed.

This book deals with those processes of destructive distillation in which the primary object is the manufacture of a solid residue, coke, of well defined character physically and chemically. In many cases this is the sole object, but during the last quarter of a century methods of manufacture have been developed which, without sacrificing the essential quality of the coke, have allowed valuable products—tar, ammonia, benzol, etc.—to be abstracted from the volatile matter evolved.

These have attracted constantly increasing attention, and of late years there has been a steady increase in the number of coking plants recovering by-products. "Modern Coking Practice" must therefore include this important branch of the industry.

During the last ten years or so the gas produced in works primarily set out for coke manufacture has been more efficiently dealt with, and the European War has brought home to this country the fact that, after all, high illuminating value is not a *sine qua non* in our lighting problems.

It is more than likely that in this country a calorific standard which can be met by coke oven gas will be substituted, and gas must therefore be considered in our list of by-products.

The main product of a coke plant is "metallurgical" or "furnace" coke, and practically the whole of it in this country is used in blast furnaces and foundries. The coke industry is thus largely dependent on the parent industry of iron manufacture.

This is shown in Fig. 1, which indicates the effect of fluctuations in the pig-iron output of the three great iron producing countries of the world. The manufacture of iron dates back to a very early period, charcoal being the fuel chiefly used in the first attempts. In 1619 Dudley endeavoured to substitute coke for charcoal, but his efforts were not seriously followed up, and more than a century elapsed before Abraham Darby reopened the question, and in 1735 successfully used coke in a blast furnace.

The invention of the steam engine in 1781, and the introduction of the puddling process by Cort in 1784, gave an impetus to the iron trade, which advanced still further on the introduction of hot blast by Neilson in 1828. Two other landmarks in the history of the iron and steel industry are the inventions of Sir Henry Bessemer in 1855 and Sir W. Siemens in 1861. These processes of steel manufacture provided ample opportunity for expansion in the pig-iron industry, but they could only deal with non-phosphoric iron.

The basic process of Thomas and Gilchrist (1879) was proved capable of removing phosphorus, and rendered available for use the enormous deposits of ore in the Cleveland district, and particularly in Germany. The whole of the German steel industry may be said to have been built on the result of this invention of a basic process.

The development of the coke industry in recent years, in sympathy with the advance in the iron industry, is shown in Fig. 1, and is emphasised by the fact that the world's output of coke has been doubled within the period covered by the chart; the present annual output throughout the world being well over 100 million tons.

Along with this development there has been an equally important change in the proportion of coke made in ovens designed for the recovery of by-products. At the present time Germany stands first in this respect, and approximately 90 per cent. of her coke is made in "recovery" ovens. In the United States the latest official returns (1919) show 56 per cent. of their coke as being made in by-product ovens.

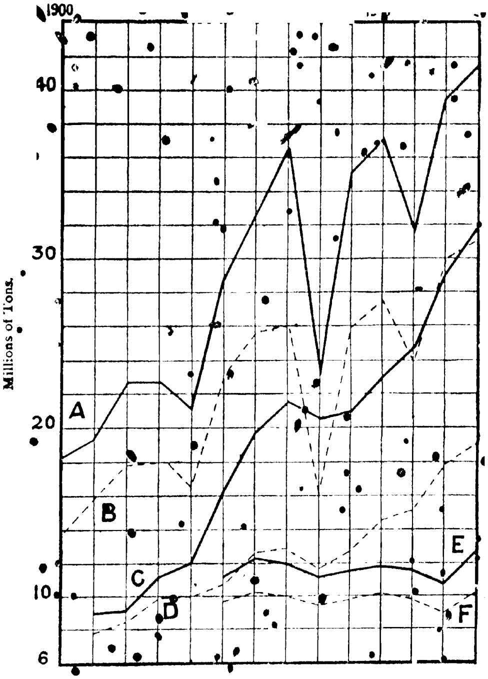

FIG. 1.

A. United States—Coke.
B. " Pig-iron.
C. Germany—Coke
D. Germany—Pig-iron.
E. Great Britain—Coke.
F. " Pig-iron.

In this country we may estimate a proportion of 75 per cent. as being made with recovery of by-products. Official figures show that in Great Britain 25,514 bee-hive ovens were in operation in 1905, together with 5,546 by-product ovens, whilst in 1915 the figures were 13,167 and 7,839 respectively. There are now about 9,700 by-product ovens in operation in Great Britain, and approximately 15½ million tons of coal

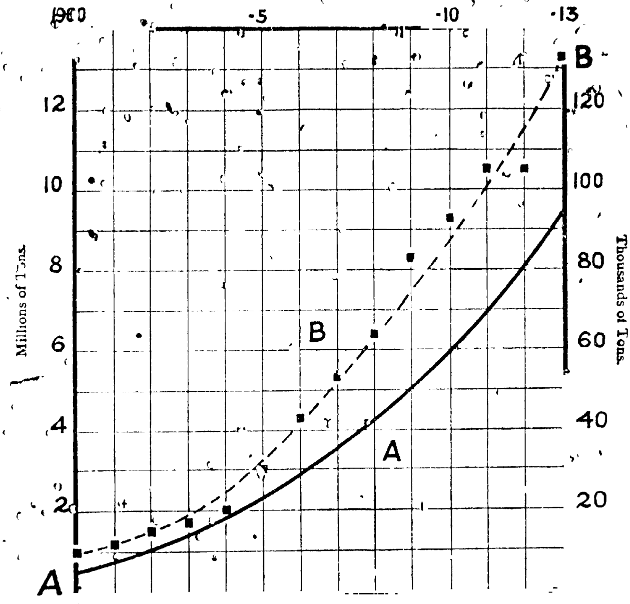

FIG. 2.

A. Coke from By Product Ovens—**Great Britain.**
B Sulphate of Ammonia—Great Britain

are treated in by-product ovens, and 5½ million tons in bee-hive ovens.

The phenomenal increase in the output of by-product coke is shown in Fig. 2, the upper curve showing the actual output of sulphate of ammonia from coke ovens, the lower curve being averaged.

CHAPTER II

CLASSIFICATION OF FUELS.

MOST of the operations in the manufacture of iron and steel are dependent on heat in some form or other. Generally speaking, anything that can be practically employed for the generation of heat may be classed as fuel.

Fuels may be divided into three classes:—

Solid.—Coke, coal, wood, charcoal, peat, lignite, etc.

Liquid.—Petroleum and its derivatives.
Coal tar and its derivatives.
Shale oils, alcohol, etc.

Gaseous.—Lighting gas, coke oven gas, producer gas, blast furnace gas, water gas, etc.

They may also be classed as:—
Organic.
Inorganic.

Dealing with the first classification, the solid fuels are the most important of the series. Apart from being the most extensively used in their raw state, they form the bases from which a great proportion of the liquid and gaseous fuels are prepared. Thus, coal tar is derived from coal, whilst all the gaseous fuels, with the exception of natural gas, are manufactured from solid fuels.

The output of coal throughout the world in 1913 was about 1,250 million tons, whilst the output of petroleum was in 1912 about 50 million tons, or only 4 per cent. of the coal output. Liquid fuel has marked advantages over solid fuels under certain conditions, but unless new oil fields are discovered and developed at an enormous rate, the world's industry will in the main depend on solid fuel.

The solid organic fuels exist in nature in many varied forms, and whilst there may be much controversy as to their manner of formation, we may take it that they are all formed from original vegetable matter changed by bacterial action, pressure, heat, etc., into the particular form in which each variety is now found

The change in physical structure and chemical composition depends largely on the geological age, and in this respect we may classify fuels from wood, which has not undergone any change, to anthracite, in which the change in physical and chemical condition is most marked.

The transition is indicated by a gradual increase in carbon content and heating value, and a simultaneous decrease in hydrogen and oxygen content, as shown in Fig. 3, which also gives an approximate idea of the yield and nature of the solid residue left on strongly heating the various classes in a closed retort.

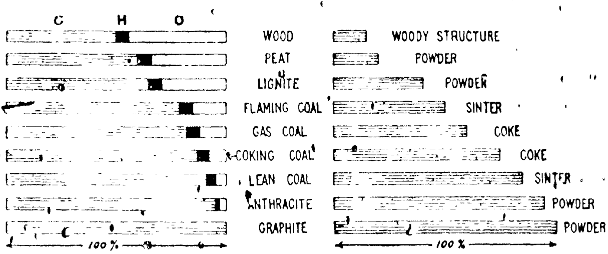

FIG. 3.

Dealing with the above fuels *seriatim*—

Wood.—This fuel is not of very great importance at the present time. It consists mainly of some form of cellulose, and when freshly cut contains a high proportion of water. Even when carefully air-dried it retains 18 to 20 per cent. moisture, and consequently its calorific value is low. It ignites very readily, however, and is useful in kindling other fuels more difficult to ignite.

Charcoal.—This is obtained by destructive distillation of wood. The yield of charcoal is low, especially if the temperature employed is high. If completely carbonised, the charcoal consists almost entirely of carbon, and possesses a high calorific value. Charcoal has been of considerable importance in iron smelting, but with very few exceptions is now replaced by coal or coke. The following by-products are obtained in modern charcoal plants :—

Gas.

Wood tar, phenols, creosote, resins, etc.

Pyroligneous acid, acetic acid, acetone, methyl alcohol, etc.

Charcoal.

Peat.—Peat is a compact fibrous substance, brown to black in colour. Its composition varies largely according to age. As in the case of wood, it is not an efficient fuel owing to the amount of water it holds, which, owing to its peculiar physical structure, even in the air-dried material, often amounts to 25 per cent. The ash content is very variable, sometimes as high as 30 per cent. In spite of the drawbacks of excessive moisture, efforts are being made to utilise peat to better advantage by carbonising it, thereby producing "peat charcoal" and quantities of tar, oils, paraffin wax, asphalt, and power gas, etc. Peat, if treated in certain forms of gas producers, is capable of giving as much as 90 lbs. of sulphate of ammonia per ton of air-dried peat, along with as much as 90,000 cub. ft. of producer gas (heat value, 150 B.Th.U. per cubic foot).

Lignite.—Lignite is a brown earthy-looking mineral, often known as "brown coal." Its physical and chemical characteristics lie between those of peat and the true coals. In the most recently formed varieties the woody fibre is not completely mineralised, and the gases on distillation show signs of acetic acid, etc. Other varieties are harder and darker in colour, having lost to a great extent any signs of woody structure, whilst the oldest varieties merge into a black pitch-like substance called "pitch coal." The ash content is extremely variable, and usually on the high side.

Lignite is not found in any great quantity in this country, but extensive beds are worked on the Continent. In Germany about one-third of the coal production is in the form of lignite, and forms the basis of a very extensive briquette industry.

Bituminous Coals.—Some of these coals on burning soften and swell like bitumen, and hence the title "bituminous" (though incorrectly) has been given to the class, which includes:—

Flaming Coals.—Long flame, rich in oxygen, non-caking.
Gas Coals.—Caking.
Caking Coals.—Strongly caking.
Steam Coals.—Only slightly caking

The first and last variety are non-caking; and do not come within the scope of this work. Some coals of the first variety, such as splint coals, are used direct in the blast furnace, whilst the names of the others indicate their special qualities. The steam coals merge through a semi-anthracitic variety to the true anthracite.

Anthracite is the most altered variety of coals, being rich in carbon, and having a shining semi-metallic lustre. It is non-caking, and extremely dense, requiring a strong blast for efficient combustion. Like splint coal it may be used direct in a blast furnace. The remaining "solid" fuel, coke, is dealt with at length in later chapters.

Liquid Fuels.—For certain definite purposes liquid fuels are extremely convenient, and possess marked advantages. They have a high calorific value, and, owing to their physical condition, can be burnt with a high degree of efficiency. Thus they can readily be sprayed or atomised, and thereby brought into more intimate mixture with the air for combustion than is possible with a bed of coal or other solid fuel.

Some are readily vaporised and, when mixed with the calculated amount of air for complete combustion, form an explosive mixture utilised in internal combustion engines.

The heavier oils are admirable fuels for boiler firing. Solid fuels require a certain depth on the grate, and unless the layer is evenly spread the air takes a selective passage, and uneven combustion takes place. This depth of fuel also entails a comparatively strong draught at the chimney, whilst variations in the load cannot always be met without excessive heat losses. Liquid fuels offer the following advantages:—

1. Economy in storage space.
2. Saving of labour in handling, etc.
3. Absence of clinkering (no ash).
4. Variable loads can readily be met.

5. Efficient combustion, hence less smoke, easily secured, and less draught necessary.
6. No boiler losses if used in internal combustion engines.

The subject of liquid fuels has increased in importance during the last few years to the modern coke industry.

It is probable that in future years the coke industry will depend largely on internal combustion engines for its market for benzol. The use of benzol for this purpose is discussed in the chapters dealing with the recovery of benzol.

Gaseous Fuels.—These are mixtures of elementary gases and vapours, some combustible, others incombustible. Of the combustible, some have considerable lighting effect such as olefiant gas, benzenes, etc., and are usually classed as "illuminants," or C_nH_m. Others are of value for their heat-producing capacity, such as hydrogen, methane or marsh gas, and carbon monoxide. The incombustible gases—carbon dioxide, oxygen, nitrogen—merely reduce the calorific value of the mixed gas, and are often known as diluents.

Analyses of Various Kinds of Gas.

	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*
Methane -	33·3	28·8		2·2	3·0	...	17·0	1·2
Hydrogen -	46·9	54·1	1·0	14·0	27·5	51·8	35·6	15·6
Carbon monoxide	5·9	5·0	32·6	23·0	11·0	43·4	28·0	20·1
Illuminants -	3·2	2·4		...		...	14·6	...
Carbon dioxide -	2·1	2·0	1·1	4·0	16·3	3·5	3·8	6·1
Oxygen - -	·4	·4				...	...	·7
Nitrogen - -	8·2	7·3	65·0	56·8	42·0	1·3	1·0	56·3

1 = coal gas;
2 = coke oven gas;
3 = producer gas (without steam);
4 = producer gas (with steam);
5 = producer gas (with ammonia recovery);
6 = uncarburetted water gas ("blue" gas);
7 = carburetted water gas;
8 = suction producer gas.

The advantages of gaseous fuels may in general be summed up as:—

1. Better regulation of temperature.
2. Oxidising or reducing atmosphere produced at will.

3. Higher temperatures can be obtained (by using regenerators).
4. Shorter chimneys required.
5. Absence of smoke under normal conditions.
6. Greater thermal efficiency if used in internal combustion engines.

Inorganic fuels are of mineral and not of vegetable origin. The chief inorganic fuels are :—

Sulphur, silicon, phosphorus, aluminium.

Sulphur acts as a fuel in the calcination of ores containing sulphur (usually in the form of iron pyrites). In some cases there is sufficient sulphur in the ore to give quite enough heat to complete the calcination without addition of coal.

Silicon.—The action of silicon as a fuel is a very important one. The success of the Bessemer acid process of steel manufacture is entirely dependent on the heat caused by the rapid oxidation of silicon. In this process some 15 tons of molten metal containing 2 per cent. silicon is run into a "converter." Air is blown through the molten metal to oxidise the silicon and other impurities. The silicon becomes oxidised to silica, and combining with other oxides passes into the slag in the form of silicates. No external heat is applied, and yet after twenty minutes' blowing the temperature is much higher. The heat caused by this oxidation is equivalent to that given off by 8 cwt. of coal burning away completely in twenty minutes.

Phosphorus acts in a similar manner in the basic Bessemer process. Pig-iron for this process should contain from 2 to 3 per cent. of phosphorus. The phosphorus becomes oxidised, and is taken up by lime and other basic materials present, forming slag.

Aluminium is now actually used as a fuel for special purposes. The heat which can be generated is quite equal to that produced in electric furnaces. Aluminium has a very strong affinity for oxygen, and if brought into contact with metallic oxides takes up their oxygen, leaving the metal in an almost pure state. If oxide of iron be used, the iron produced reaches such a temperature that if poured into blow holes in castings, etc., it actually fuses the surface of the casting, becoming welded to it. For this purpose the aluminium must be in fine granular state of division, and must be mixed in definite proportion to the oxide, according to the formulæ :—

$$3FeO + 2Al = 3Fe + Al_2O_3$$
$$Fe_2O_3 + 2Al = 2Fe + Al_2O_3$$
$$3Fe_3O_4 + 8Al = 9Fe + 4Al_2O_3.$$

This mixture is placed in a crucible covered with a smal proportion of a priming mixture of aluminium and barium peroxide in the following proportion :—

$$3BaO_2 + 4Al = 3Ba + 2Al_2O_3.$$

This priming mixture, on being ignited by means o magnesium ribbon, brings about a local heat sufficient to start the main reaction.

Other metals of high melting point, whose oxides are very refractory, may be reduced by the above process, such as chromium, tungsten, manganese, etc.

CHAPTER III

IMPURITIES IN COAL

THE impurities in coal are of great importance in the coking industry. The chief are ash, sulphur, and chlorine, principally in the form of sodium chloride or salt. Ash is composed of incombustible mineral matter left as a residue on burning completely coal, coke, etc. Its composition is variable, consisting chiefly of silica, 30 to 60 per cent.; alumina and oxide of iron, 35 to 55 per cent.; lime, 5 to 10 per cent., along with smaller quantities of magnesia and alkalis. Some of these substances are combined with sulphuric acid (3 to 8 per cent.) as sulphates.

An excessive ash content is objectionable in fuels for steam raising, as the clinker formed obstructs the passage of the air for combustion, and carries away with it unconsumed carbon in the form of cinders. The importance of the ash content is seen in the coking process, in which most of the coke sold must contain less than 12 per cent. of this impurity. The mineral matter is not removed in coking, and is therefore concentrated in a lesser amount of coke.

Roughly speaking, a coal giving 7 per cent. ash will make coke containing 10 per cent. As far as the blast furnace is concerned, oxide of iron in the ash is not detrimental, but the silica requires additional limestone to flux it. The colour of the ash is a fairly reliable indicator of the proportion of oxide of iron, a deep red colour denoting a high content of iron. As the iron compounds spring from sulphide of iron in the coal, a red ash would indicate also a high proportion of sulphur in the coal, this being a most objectionable impurity.

Sulphur exists in coal in four conditions:—

1. As iron pyrites (coal brasses, iron bisulphide).
2. As sulphate of lime or alumina.
3. In an organic form, combined with carbon or hydrogen.
4. In rare cases as free sulphur.

Of these, that which occurs in the form of pyrites is the most injurious, since, on combustion of the fuel, its sulphur becomes oxidised to sulphur dioxide, which in presence of moisture becomes sulphurous acid, and eventually, by further oxidation, sulphuric acid. This has an intensely corrosive effect on any iron or steel with which it may come in contact, especially the cooled surfaces of economiser tubes or other parts on which moisture can condense, thus absorbing more readily the acid fumes.

The sulphur in the form of sulphate is not injurious, except that it adds to the mineral constituents or ash of the coal.

Iron pyrites is also objectionable in this manner. On burning the fuel the oxide of iron formed becomes reduced to ferrous oxide, which readily combines with the silica and alumina of the ash, forming a fusible mass known as clinker, the ill effects of which have already been mentioned.

In coking practice some of the sulphur passes off with the volatile matter, part is liberated by the action of steam on quenching the coke, and part remains in the coke. The following gives an idea of the distribution of sulphur in carbonising Durham coking coal[1]:—

(*a*) Left in coke	72·53	per cent.
(*b*) In gas	19·70	„
(*c*) In tar and liquor	7·64	„

Chlorine is usually found in coals as the sodium compound, common salt, the alkali being an important factor in the corrosion of coke oven walls. Authorities are not unanimous as to the manner in which refractory materials are affected by alkalis. In coke oven practice, the oven walls may be affected by the total soluble salts, not necessarily chlorides; by the deposition of carbon in the pores of the brick; by the volatilisation of iron in the form of chloride along with deposition of iron, and by the decomposition of sodium chloride.

Professor Cobb, of Leeds University, gives the following explanation of the action of salt.[2] If coal containing more than ·05 per cent. NaCl is charged into an oven the walls suffer, although a larger amount does little damage if the temperature is low. Below 800° C. sodium chloride volatilises, and, penetrating into the brickwork, combines with its substance. Near the face of the brick in contact with the coal the temperature is not sufficiently high to cause fusion, but as we approach the face of the brick directly heated by the flue gases, and accordingly at an increased temperature, we arrive at a point at which the sodium silicate or sodium—alumina—silica compounds begin to fuse. This portion becomes porous, honeycombed, and weak in structure, setting up disintegration often severe enough to cause the face of the brickwork to fall away in thick layers. The authors' experience tends to confirm this theory, but leads them to believe that a high iron content in the coal is also deleterious, and would serve to intensify the corrosive action if iron and chlorine were present together.

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[1] Andrew Short, Society of Chemical Industry, 1907.

[2] "Refractory Materials and Salty Coals," Prof. J. W. Cobb, Coke Oven Managers' Association, 1916.

Various observers have found a definite increase in alkali and iron content in the corroded portions of brickwork.

	Original	Corroded.	Original.	Corroded.	
Alkalis - - -	1·22	4·63	...	...	Schrieber
K_2O - -	1·44	4·33	Fe_2O_3 1·15	3·71	Blasberg
K_2O - -	1·06	4·60	Fe_2O_3 1·12	4·52	Blasberg
K_2O - - -	·40	4·35	..	. .	Holgate
Na_2O - -	·25	2·65	...	...	Holgate
...	..	. . .	Fe 1·18	2·51	Simmersbach
NaCl and KCl	·7	8·4	.		Chaney
Alkalis - -	·75	4·5	Fe_2O_3 1·50	7·50	Byrom

The above are all cases of corrosion, and show that salt plays a large part in the destructive effect. Coals with less than ·05 per cent. NaCl do not appear to have any serious destructive action. The quality of the washing water should not be overlooked, as, in many cases, such waters contain excessive amounts of salt, and often very considerable quantities of sodium sulphate. The chlorine liberated by the decomposition of salt combines with ammonia, forming ammonium chloride which is volatilised in the oven, and if excessive may give rise to a thickening of the tar in the mains, and this is likely to be aggravated if magnesium chloride is present, which is occasionally the case. In some processes of ammonia extraction there is a risk of some of this ammonium chloride being carried to the lead-lined saturator, in which hydrochloric acid in small quantity might be liberated, thereby corroding the lead lining, and in these cases special precautions are taken to remove the ammonium chloride previous to this stage. Corrosion in boiler tubes has in some cases been traced to chloride.

CHAPTER IV

COAL WASHING

COAL, as it comes from the mine, is always contaminated to a greater or less degree with impurities of various kinds which, though practically unavoidable, lessen considerably the value of the coal for most purposes. Some of these, such as siliceous and calcareous shaley matter, and pyrites, are actually associated with the coal itself, whilst others get into the coal from the roof and floor during the operation of mining. It is obviously of material benefit, both to seller and consumer, if these foreign substances can be removed in some way, and the method usually adopted is to treat the coal in some form of washing apparatus whereby it is rendered more or less free from the above-mentioned materials. There are several types of washers in general use, and it will be the purpose of this chapter to describe a few of the washers used in this country.

There are many points to be dealt with in considering the washing of coal, given an effectual type of washer. There is the cost in the first instance, then the physical nature and condition of the coal itself, some coals being much more friable than others, producing more "smalls" in the mining, then the specific gravity of the coal and impurities, shale, pyrites, etc. Again there may be several kinds, such as bone coal, cannel, and shaley coal, in the same seam, and the specific gravity of these will probably differ appreciably from that of the main bulk. The great point to be aimed at is, of course, to wash the coal as completely as possible, removing a maximum amount of impurity, with a minimum amount of coal passing with the dirt. The analysis of representative samples of the material before and after washing will give the percentage elimination of mineral matter and sulphur. It must be remembered that even the cleanest piece of coal which can possibly be selected is by no means absolutely free from ash; in fact, some coals picked as clean as possible will contains 10 per cent. or more of mineral matter. Now no amount of washing can possibly remove such substance, because it is ingrained in the substance of the coal itself, and no doubt got there during the formation of the seam ages ago. In fact much of it is due to the ashes of the plants from which the coal was formed. The amount of coal passing away with the dirt should be carefully watched, and this may be readily done by taking into account the specific gravity of the coal, and of the dirt separated from it.

For the moment assume that the specific gravity of the coal is 1·25, and that of the shale 2·2, and pyrites 5·0. It is evident that by making a solution of calcium chloride (or other suitable substance), with a specific gravity of 1·3 to 1·35, we shall have a liquid in which shale and pyrites will sink and coal will float. Hence, if a weighed portion of the dirt be taken (500 or 1,000 gm.), placed in the above solution, and well stirred up, the coal which floats can easily be skimmed off, placed in a filter paper, washed free from calcium chloride, dried and weighed, giving the percentage of coal in the dirt by a simple calculation. This method, whilst useful for laboratory tests, is much too expensive to be adopted on a commercial scale. In coal washing, water is used, and we rely on the difference in the velocities at which the various substances sink in water. Particles sink in water at a gradually increasing speed until they attain a constant maximum velocity, which may be approximately determined by Rittinger's formula:—

$$\text{Velocity in feet per second} = 1{\cdot}28\sqrt{D}\,(d-1).$$

D = Diameter of particles in inches.

d = Specific gravity of material.

1·28 = Constant obtained as a result of numerous experiments.

The specific gravities of substances connected with coal washing may be taken as:—

Coal	1·25 to 1·60 (average 1·3)
Shale	2·2
Shale with pyrites	3·4
Pyrites	5·0
Felspar	2·4
Quartz	2·7

From the above formula we see that with similar material a large piece will sink in water at a quicker rate than a smaller, and pieces of coal, shale, and pyrites of one inch diameter will attain maximum velocities of ·76, 1·41, and 2·56 ft. per second respectively. Thus for particles of equal size the velocities are approximately in the ratio of 1, 2, and 4. However, as the particles contained in the "run of mine" coal are of varied sizes mixed indiscriminately, we often find large pieces of coal and small pieces of dirt in close proximity and we can calculate that the falling velocities of a two-inch piece of coal and a half-inch piece of shale are identical.

The principle of coal washing is to treat the coal with water in such a manner that the impurities sink to the bottom of the vessel whilst the coal is kept above the impurities, and it is obvious that in the above extreme case this would be impossible.

It is therefore of importance that the coal be correctly screened, and as a rule the diameter of the largest particles should not exceed four times the diameter of the smallest.

In most washeries the coal is screened before washing in accordance with the above principle, whilst in some cases the equivalent result is secured by screening after washing and rewashing the smallest sizes. There are several types of washers in use, and in all of them the coal is agitated in a good supply of water, the agitation being brought about by various means, viz.:—

(*a*) The flow of the water itself.
(*b*) Travelling belts and scrapers, etc.
(*c*) Revolving arms.
(*d*) Pulsation of the water by pistons, or compressed air.

The simplest type of washer is the trough washer (Fig. 4). It consists of a trough A, varying in length from about 40 to 100 ft., about 3 ft. wide and 15 in. deep. At intervals movable dams (B) are placed. The trough is set at an inclination sufficient to cause the necessary agitation of the coal for the efficient separation of the heavier shale and pyrites. The dirt is caught by the dams B, and at intervals is run off from the side of the trough through the sluice C, after removing the dams B. The washed coal passes over the screen D, the water passing into the channel (G). These washers are often worked in pairs, one being cleaned out whilst the other is engaged in the washing operation.

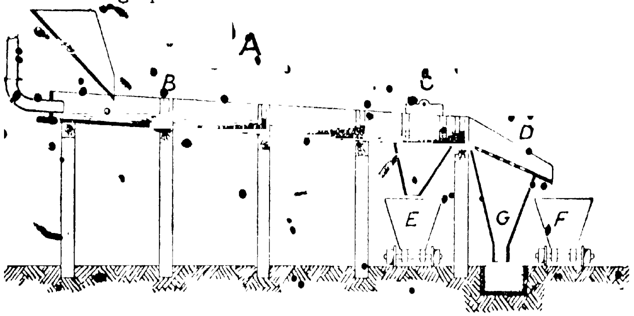

FIG. 4.—Trough Washer.

The Elliott washer (Fig. 5) utilises the principle of the trough, but the dirt is removed continuously by means of a scraper chain. The working of this washer is more under

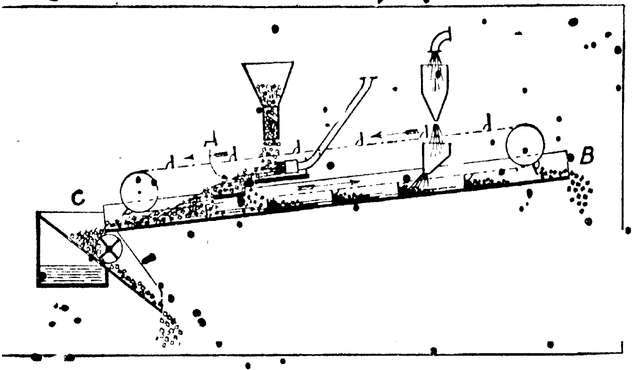

FIG. 5.—Elliott Washer.

control, and can be regulated by altering the inclination of the trough and the speed of the scraper chain to suit the various classes of coal dealt with. A rough or primary washing is carried out by dropping the unwashed coal on to a preliminary washing table (A). The rougher or larger sized dirt drops between the two plates, and is immediately removed by the scrapers, whilst the lighter coal is swept forward. This

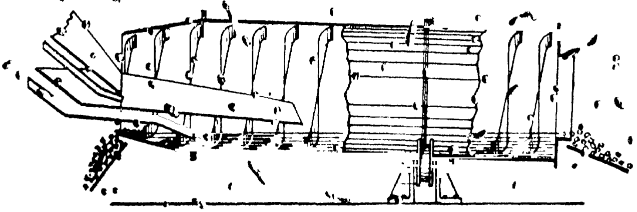

Fig. 6.—Blackett Washer.

reduces the work in the washing trough and ensures more efficient working. The washed coal passes over a primary drainer C to get rid of the bulk of the water.

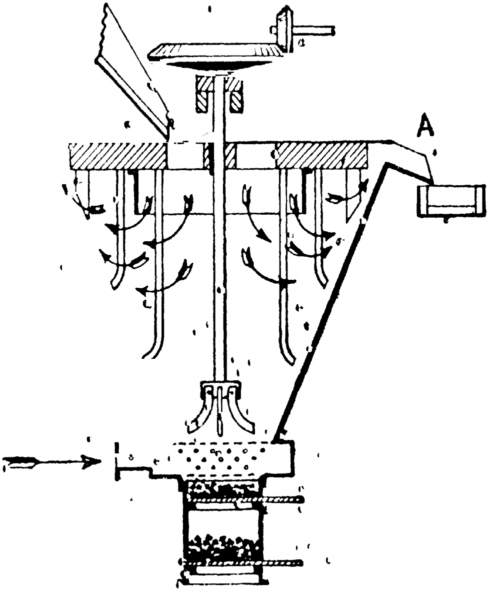

Fig. 7.—Robinson Washer

The Blackett washer (Fig. 6) is a type of washer in which the agitation is brought about by means of a revolving barrel, set at an inclination to suit the size of coal washed, etc. At the same time a shallow worm attached to the inside of the barrel gradually forces the heavier "dirt" to the upper end of the barrel, where it is discharged, whilst the true coal is washed over the worm, and delivered at the lower end on to a drainer.

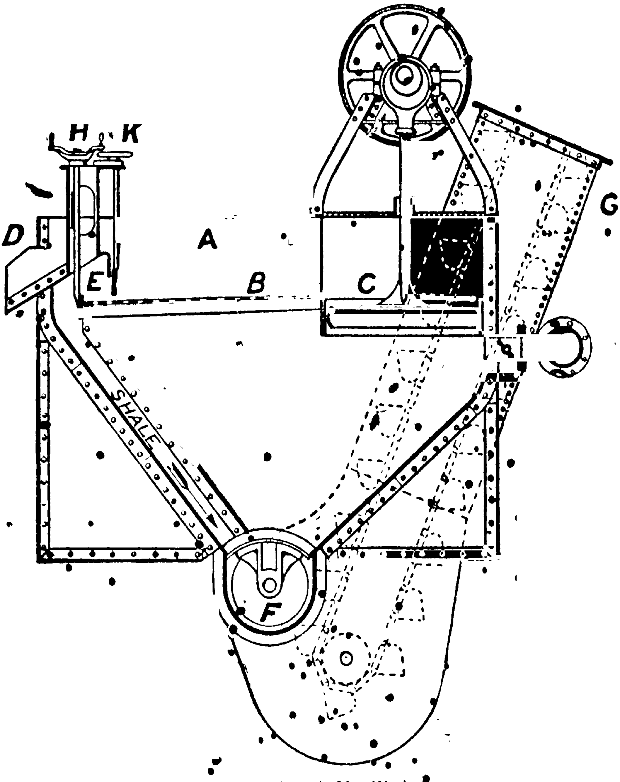

FIG. 8.—Coppée Nut Washer.

The Robinson washer (Fig. 7) consists of a funnel-shaped chamber, the contents of which are kept in motion by means of revolving arms. The water is pumped in at the bottom of the cone, and flows over the edge of the funnel at A, carrying

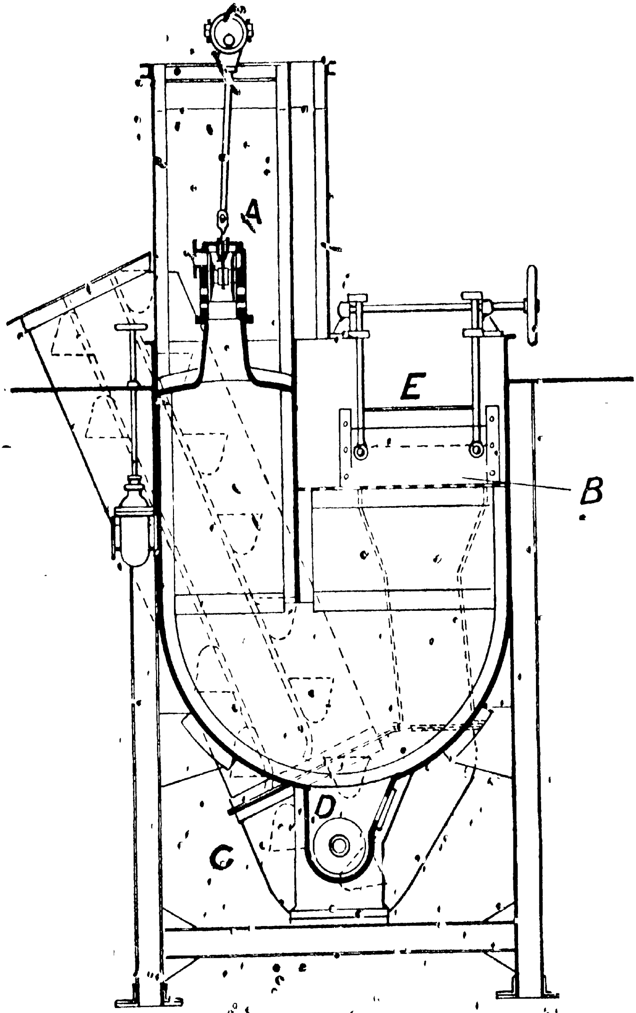

FIG. 9.—Baum Washer.

the washed coal with it. The dirt settles to the bottom, and is removed by means of the double-doored arrangement shown in sketch.

In the fourth class of washer the coal is agitated by currents of water set up by pistons, plungers, compressed air, etc. This type, known as a "jig" washer, though more expensive than the former machines, is a very efficient one, the type shown in Fig. 8 (Coppée nut washer) being a good example. In this the coal is fed into the section A, being supported by the sieve B. The plunger C causes an alternate ascent and descent of the water under the sieve. The coal is thus well agitated, and the lighter coal is forced to the top whilst the heavier dirt settles on the screen. The washed coal is carried over the sluice D by the washing water, whilst the dirt is removed at E, by means of sluices controlled by the shale valves H and K. Various mechanical arrangements such as the screw conveyor F, elevator G, etc, are used according to the type of washer to facilitate the removal of the dirt. In the Baum washer (Fig. 9) the principle of the jig is also utilised, but the pulsation of the water is brought about by means of compressed air, at a pressure of about 2 lbs per square inch. This is admitted and released by the specia slide valve A, shown in detail in Fig. 10.

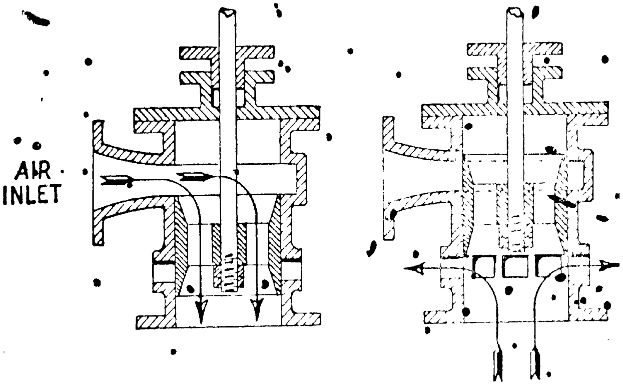

FIG. 10.—Air Valves, Baum Washer

The result of this particular arrangement is a rapid up-stroke in the washing action, but a slower down-stroke, allowing a longer interval for the materials to settle, according to their respective gravities. The rough dirt is run off as desired by means of the controlled sluice B to the elevator C direct, whilst the fine dirt falls through the screen to the bottom of the washer, whence a screw conveyor D directs it to the dirt elevator. The clean coal is washed over the outlet E. In this washing system the coal is washed first, then screened, and the smallest sizes rewashed. For very small sizes of slack a felspar washer is extremely serviceable, being almost automatic in its action. In this type (Fig. 11, Coppée felspar washer), the mesh of the screen A is large enough to allow all the particles of coal and dirt to pass through. A bed of felspar of a size too large to pass through the meshes is placed on

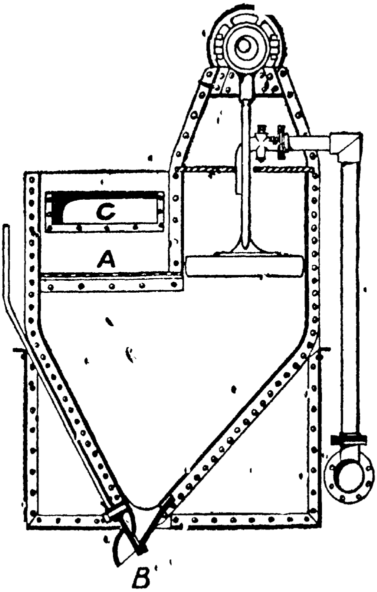

FIG. 11.—Coppée Felspar Washer.

the screen, and on agitating the water and admitting the fine slack the materials tend to settle in the following order:—

Coal	- - - -	Specific gravity 1·3
Felspar	- - - -	,, 2·4
Dirt	- - - -	3·4 to 5·0

The dirt thus ultimately descends to the screen, passes through it, and is collected at B. The washed coal during the "suction" period is prevented from passing through by the bed of felspar, and during the up-stroke is washed over the gate C, and collected.

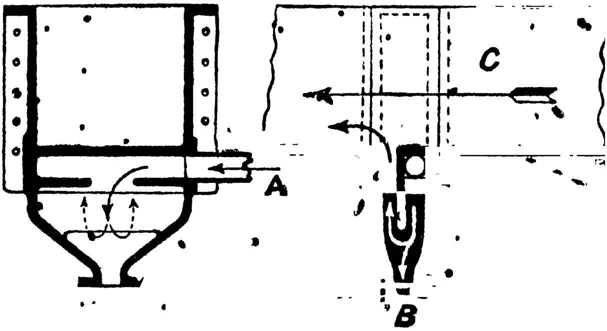

FIG. 12.—Rhéolaveur.

In jig washers the length of stroke and the number of pulsations per minute is regulated according to the size of particles treated, a slower speed and longer stroke being used for the larger sizes.

An interesting development in coal washing is shown in Fig. 12. This type of apparatus, known as the Rhéolaveur, is the invention of Messrs Habets & France, and is used in conjunction with a trough washer. Water is admitted under sufficient pressure at A, and the stream is divided into two currents by the baffle plate. The ascending current is at a velocity sufficient to counterbalance the falling velocity of coal, but is not sufficiently powerful to lift the particles of shale, etc. The shale thus sinks, comes under the influence of the descending current, and passes through the outlet B with an adjustable opening. The washing is thus continuous. The number and disposition of these Rhéolaveurs in a trough is regulated by the quantity of coal dealt with, and the relative amount of dirt to be removed. In some cases the shale separated at the lower end of the trough is rewashed in a secondary trough with Rhéolaveurs specially adapted for this purpose.

CHAPTER V

SAMPLING AND VALUATION OF COAL, COKE, ETC.

In making a physical or chemical examination of coal or other material, it is obviously of first importance that a representative sample of the substance be obtained. It is impossible for one or two small pieces, say of coal, to represent fairly an average composition of any particular seam; they may be either too clean (which is the more likely) or too shaley. At the least, several barrows full of coal should be taken and roughly broken down, in the first instance, on a clean surface, preferably iron or steel. The heap should then be well turned over and mixed up, spread out, and divided crosswise into quadrants. Opposite sections should then be taken away, thus halving the total sample. The remainder is crushed smaller, again thoroughly mixed up and quartered, rejecting half. This is repeated until the sample has been reduced to a reasonable quantity, say 10 or 12 lbs., which should then be crushed, if necessary, until all passes through a sieve of about $\frac{3}{16}$-in. mesh. From this a small sample is taken after thorough mixing, crushed somewhat smaller, and a final sample of 1 or 2 oz. crushed so as to pass an 80 sieve. It is then ready for analysis, and is a fair average sample of the material.

Specific Gravity of Coal, Coke, Shales, etc.—The specific gravity of solids insoluble in water, such as coal, shale, pyrites, etc., may be readily determined, given a moderately accurate balance and set of weights. The test may be done on small lumps or on the powdered material. If lumps are used, then several determinations are desirable on different pieces, taking the average result as the true figure. If the powdered substance is used, only one, or at the most two, determinations will be needful. In these determinations the principle of

Archimedes is made use of, namely, that when a body is submerged in a liquid it loses weight equal to the weight of liquid displaced by it. In an actual determination, if a small lump is being used, it is suspended by a loop of horse hair from one end of a balance and carefully weighed. It is then immersed in water in a glass vessel, supported on a wooden bridge (Fig. 13), and again weighed.

A considerable decrease in weight will be noticed, due to the operation of the above-named principle. If W be its weight in air, and w its weight in water, then the specific gravity of the solid is—

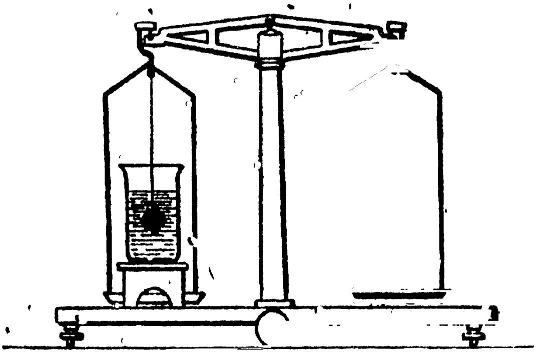

FIG. 13.—Specific Gravity Balance.

$$\frac{W}{W-w}$$

Example:—

Weight of sample in air	-	16·342 gm.
Weight of sample in water	-	4·098 „
Weight of water displaced	-	12·244 gm

$$\text{Specific gravity} = \frac{16·342}{16·342-4·098} = 1·334$$

In the case of a powdered sample it is, of course, impossible to use this method, and the use of a specific gravity bottle is necessary. This is a small bottle (Fig. 14) fitted with an accurately ground stopper, through which a fine hole is drilled lengthwise, to enable the bottle to be completely filled with water or liquid, which is not easily done with the solid stopper. It is first carefully filled with distilled water and the stopper inserted. After carefully wiping off all superfluous liquid from the outside, the bottle is weighed. A portion of the water is then emptied out, and 1 gm. of the powdered coal or other mineral is placed carefully in the bottle, and well shaken to thoroughly wet the material. This is not at all easy in the case of the coal, and great care should be taken that all air bubbles, which are readily enclosed by coal dust, are completely got rid of, otherwise the result will be inaccurate.

FIG. 14.
Specific Gravity Bottle.

An example will best illustrate the calculation of the result :—

Weight of specific gravity bottle filled with water - - - - - - 73·498 gm.
Weight of bottle containing water and 1 gm. of coal - - - - 73·745
(*i.e.*, weight of bottle and water left in after displacement by the coal - 72·745 „)
∴ Weight of water displaced = 73·498 − 72·745 or ·753 gm.
∴ Specific gravity = $\frac{1}{\cdot753}$ or 1·328.

The specific gravity of liquids may be obtained either by use of some form of hydrometer or by means of the specific gravity bottle. The Twaddell hydrometer (Fig. 15) is in common use in this country. The readings obtained by its use are readily converted into specific gravity by multiplying by 5 and adding 1,000 (taking the specific gravity of water as 1,000), thus :—

If the hydrometer floats at 65°, then taking specific gravity of water as 1,000, the specific gravity of sample would be

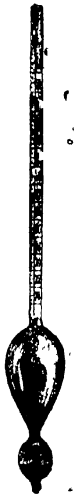

FIG. 15. Twaddell Hydrometer.

(65 × 5) plus 1,000, or 1,325, or taking specific gravity of water as 1, the specific gravity of the sample would be 1·325.

Conversely, if the specific gravity is given to three decimal places, neglect the decimal point, subtract 1,000, and divide by 5. The result will indicate degrees on the Twaddell scale. A saturated solution of calcium chloride at a temperature of 15° C. contains 40·66 per cent. of the salt, and has a density of 82·2° Tw., which is equal to a specific gravity of 1·411. If the specific gravity bottle be used, it is first carefully weighed empty and dry, then filled with distilled water at about 15° C. and weighed again. The water is emptied out and replaced by the liquid whose specific gravity is required (the temperature being the same as that of the water), carefully filled and weighed. The results give the relative weights of equal volumes of water and the liquid to be tested. From this the specific gravity is readily obtained. Thus:—

Weight of bottle filled with water -	45·438 gm.
Weight of bottle (empty and dried) -	20·652 „
Weight of water - - - -	24·786 „
Weight of bottle filled with liquid -	50·672 gm.
Weight of bottle (empty and dried) -	20·652 „
	30·020 „

Hence the specific gravity of the liquid is:—

$$\frac{30·020}{24·786} \text{ or } 1·211.$$

It is often important to determine the amount of moisture in coal, as even that which is freshly mined contains appreciable amounts, usually from 2 to 5 per cent. If the mineral has been exposed to wet weather, it is very likely to contain more. Some coals will take up and retain more moisture than others, owing to their physical condition. To ascertain the amount, a fair average sample is taken and crushed down to not less than ¼ in. in size, a weighed quantity taken (say 100 or 250 gm.) and dried for three or four hours at a temperature of 100° C. in a water bath, allowed to cool, and the loss ascertained by weighing.

If the sample be reduced to a very fine condition, it is liable to lose other constituents than moisture on prolonged heating, even at 100° C. Also pyrites may become oxidised. A safer plan, and one which is useful as a check is to expose a weighed portion under a bell-jar desiccato for at least twelve hours, and weigh the loss due to moisture which is taken up by the sulphuric acid or calcium chloride used in the desiccator. In this way there is not the same danger of driving off easily volatile substances along with the moisture.

Estimation of Ash in Coal and Coke.—The amount o ash contained in coal or coke may be determined by taking a weighed portion from the finely powdered sample and igniting it carefully, either over the straight Bunsen flame or preferably in a gas muffle, cautiously and gradually a first to avoid loss by decrepitation, increasing the heat unti finally a good red heat is reached, combined with a plentifu air supply. It will be found that coal burns off more quickly than coke. In the case of hard-burnt coke the ash should be weighed until it ceases to lose weight.

When a coal is completely burnt off it ceases to glow but it is not easy to judge a coke in this way, and especially if the ash is high, as this protects the small particles of hard coke from oxidation, so that occasional careful stirring i desirable. Small porcelain crucible lids, about 1¾ in. in diameter, are very suitable for burning off coke or coal. I will be found that if platinum vessels are used, the meta becomes brittle and cracks after a time, probably due to prolonged heating in contact with carbon. After all th carbonaceous matter has been oxidised, the ash is carefully brushed out of the dishes, when cool, on to the balance pan and weighed. Thus, if 1 gm. of coal have an ash weighing ·087 gm., percentage of ash in the coal would be 100 × ·087 or 8·7 per cent.

The colour of the ash should be noted. Since all th mineral matter which is in the original coal, with the excep tion of some of the sulphur, remains in the coke, it is obvious that the ash in the coke is higher than that in the coal from which it is made. For instance, if a coal contains 7 per cent. of ash, and yields 70 per cent. of coke, the ash in the latter will be $\frac{7}{70} \times \frac{100}{1}$, or 10 per cent.

Estimation of Volatile Matter.—The volatile matter in coal is that portion which is driven off or volatilised, when the fuel is heated out of contact with air. It usually includes some portion of the sulphur which is present in the coal, and it is generally taken that about 50 per cent. of the sulphur is thus driven off. The volatile matter is estimated by heating a weighed portion in a closed crucible, without admission of air. One or two grams may be taken and gently heated over a Bunsen flame until smoke ceases to issue from the crucible; it is then quickly transferred to a muffle furnace at a good red heat, the furnace door closed, and the heating continued for two and a half or three minutes, after which the crucible and contents are removed, placed under a bell-jar desiccator till cool, and then weighed. The loss represents volatile matter, and the residue, coke, *i.e.*, fixed carbon, ought not to contain any volatile matter, but usually a slight amount is present, say from ·5 to ·8 per cent.

Estimation of Sulphur.—The sulphur in coal and coke may be estimated by—

(*a*) The lime method, or
(*b*) Eschka's method.

(*a*) Weigh off carefully 1 gm. of the fuel and 1 gm. of pure lime, on which a blank estimation for sulphur has been done. Moisten to a fairly stiff paste with distilled water, and mix up intimately with a spatula or glass rod, being careful to avoid any loss. Dry slowly in a water bath or under a hot plate, and place in front of the muffle furnace for about half an hour, giving free access of air. Afterwards heat strongly in the muffle for a further half hour. By this treatment the sulphur in the fuel becomes converted into sulphate of lime. After removal from the furnace and cooling, the mass is transferred completely to a small beaker and moistened with distilled water. Sufficient hydrochloric acid, containing a few drops of bromine (both free from sulphur), is added to dissolve the calcium sulphate and excess of lime. The solution is then boiled and filtered. After washing the filter paper, the filtrate is boiled, and while boiling a hot solution of barium chloride is added cautiously. This reacts with the calcium sulphate in solution, forming barium sulphate, which is precipitated as a heavy, white powder.

It should be allowed to settle completely, which will probably take a few hours, then filtered off through Swedish paper, taking care that none passes through the pores of the filter. It is finally washed and dried, ignited and weighed as barium sulphate (which contains 13·73 per cent. of sulphur). From this the percentage of sulphur in the fuel may be readily obtained. Thus 1 gm. of coal yielding a precipitate of barium sulphate weighing ·12 gm. would contain ·12 × 13·73 gm. of sulphur, equivalent to 1·647 per cent.

(*b*) In Eschka's method 1 gm. of the fuel is taken, finely divided and mixed in a shallow dish with about 1 gm. of pure calcined magnesia, and about ½ gm. anhydrous sodium carbonate. The mixture is then covered with a thin layer of magnesia (about ½ gm.). As in the case of the lime method, a blank determination of the sulphur in the material used must be made, and the weight of barium sulphate obtained (if any) deducted from the total weight obtained. The dish and the contents are placed in front of the muffle furnace for about three-quarters of an hour, allowing free access of air, then finally heated somewhat more strongly for fifteen or twenty minutes. After removal from the muffle and cooling, the contents of the dish are carefully transferred to a beaker and hot distilled water added, with a few drops of bromine and hydrochloric acid, boiled and filtered clear of insoluble residue. After washing, the solution obtained is boiled up and barium chloride added, as in the lime method, to form a precipitate of barium sulphate. The subsequent operations are carried out as in method (*a*). In this method the sulphur of the fuel becomes converted into the sulphates of magnesium and sodium, both of which are soluble in water. The bromine oxidises any sulphides that may have been formed to sulphates as in (*a*).

When coal is heated in a closed oven or retort, as in the various methods of coking, it is said to be subjected to destructive distillation. It is broken up into a number of products, solid, liquid, and gaseous, the proportion of which largely depends upon the temperature employed and the nature of the particular coal which is being carbonised. If the temperature is high the products are mainly gaseous. On the other hand, if the temperature is low, heavy tarry liquids are formed, which retain various solid substances either in solution or suspension. Under normal conditions of coking the volatile hydrocarbons are almost completely expelled. A well-burnt coke ought not to contain more than 1 per cent. of volatile matter. Along with the volatile substances are other gaseous products, such as cyanogen, ammonia, carbon dioxide, sulphuretted hydrogen, and free nitrogen.

All coals contain more or less nitrogen, the content of nitrogen in British coals varying from about ·8 per cent. in some cases to nearly 2·0 per cent. in others; and other conditions being equal, the yield of ammonia in any coal distillation in general varies with the nitrogen content of the coal.

If, however, a coal is only partially gasified, as in the gas retort or coke oven, a portion of the nitrogen remains in the coke in a rather stable condition, and is thus, under ordinary conditions, unavailable for the production of ammonia.

The proportion of nitrogen converted into ammonia has been investigated by various observers, and results of four investigations are given below :—

	1 Foster (English Coals)	2 Knublauch (German Coals)	3 M'Leod (Scotch Coals)	4 Short (Durham Coals)	Average.
	Per Cent	Per Cent.	Per Cent.	Per Cent	Per Cent.
In coke	48·68	50·0	58·3	43·31	50·5
As cyanogen	1·56	2·0	1·2	1·43	1·5
In tar	35·26	30·0	3·9	2·98	3·5
In gas (free N)			19·5	37·12	29·5
As ammonia	**14·5**	**13·0**	**17·1**	**15·16**	**15·0**

Under modern conditions coke ovens attain a higher state of efficiency than the above, and 16 to 17 per cent. of the nitrogen content of the coal has been recovered as ammonia.

The yield of ammonia at coke works is affected by several factors, the main being (*a*) temperature of coal distillation; (*b*) duration of distillation period; (*c*) presence of extraneous bodies; (*d*) exposure of gases to heat.

(*a*) With reference to the temperature of distillation, experiments by L. T. Wright gave the following results:—

Temperature.		Yield of Sulphate of Ammonia.		
Very low	- - -	16·4 lbs. per ton of coal.		
Low	- - -	18·4	„	„
Moderate	- - -	20·4	„	„
High	- - -	19·1	„	„

Mayer and Altmayer also obtained the following results with different classes of coal :—

Temperature.	Lbs. Sulphate of Ammonia per Ton of Coal			
	1.	2.	3.	4.
600° C. - -	12·6	8·4	6·8	6·1
700° C. - -	23·4	16·6	14·7	7·1
800° C. - -	25·8	21·3	16·2	9·5
900° C. - -	21·7	20·6	17·3	8·9
1,000° C. - -		...	15·7	
°/₀ N in coal -	1·13	1·32	99	·51
°/₀ Ash in coal -	4·31	8·63	21·08	11·61

From the above we may gather that at low temperatures the yield of ammonia is relatively low, gradually improving as the temperature rises, and then on reaching a temperature between 800° C. and 900° C. we arrive at a point at which the higher temperature is beginning to have an effect in dissociation, and our yield of ammonia begins to decrease.

(*b*) As we have already seen, some 50 per cent. of our nitrogen is left in the coke in a well-fixed condition. Prolonged heating tends to expel some of this, but unless other conditions assist the union of this free nitrogen with hydrogen, an appreciable increase of ammonia yield is not apparent, and the diminution of the output of the oven or retort might more than counterbalance any gain from ammonia.

(c) Experiments on this point go to prove that the addition of certain outside bodies to a mass of coal during distillation has a marked effect on the ammonia yield, some bodies being favourable, others being decidedly unfavourable.

Thus an addition of free nitrogen, calcium carbonate, lime, or steam appears in each case to increase the yield, whilst the reverse effect is noted on adding either of the following bodies: carbon dioxide, ferrous carbonate, ferrous oxide, or ferric oxide. The beneficial action of calcium carbonate would be largely neutralised by the corrosive action of the calcium oxide on the expensive brickwork of the retort or coke oven, shortening the life very materially. The advantage of a steam atmosphere is best seen in the Mond gas producer, in which a yield of about 90 lbs. of sulphate of ammonia can be obtained as against, say, 24 lbs. from similar coal in coke ovens.

The deleterious effect of iron compounds on the ammonia yields should also be noted. In this respect the amount of ash in a coal, and more especially the composition of the ash, may have a marked effect on the ammonia yield.

(d) Exposure of gases to heat. If we pass a current of dry ammonia gas through a heated tube decomposition takes place. Ramsay and Young found that, with a porcelain tube filled with pieces of porcelain, ammonia was decomposed to the extent of 69·5 per cent. at a temperature about 820° C.; whilst with an iron tube filled with the same material 100 per cent. of the ammonia was decomposed at a temperature of 780° C. These results appear contradictory to our previous statements, but in this we must consider the protective influence of other bodies. In coke works practice we have a considerable amount of water vapour evolved with the gases, and taking this into account we can calculate that in coke works practice each cubic foot of ammonia evolved is accompanied by 170 cub. ft. of fixed gases, water vapour, etc. Considering the velocity at which these gases are withdrawn from the ovens, there is very little time for dissociation to take place. At the same time it is an undoubted advantage to keep the exit passages as cool as possible, and in good coke oven practice this point is considered, in arranging the side flues, so as not to unduly heat the crown of the oven.

Estimation of Nitrogen in Coal and Coke.—The method most suitable is that known as the Kjeldahl process, which consists in heating the finely divided material with concentrated sulphuric acid in presence of manganese dioxide. The effect is to destroy all the carbonaceous and organic matter, and at the same time to convert the whole of the nitrogen into ammonium sulphate. On then adding a moderate excess of sodium hydrate solution, and distilling into a standard solution of sulphuric acid, the amount of ammonia liberated is calculated from the quantity of sulphuric acid neutralised. As to quantities, 1 gm. of coal is a suitable amount to use. Heat with 25 to 30 c.c. of strong sulphuric acid for half an hour. Then add 5 gm. of manganese dioxide and allow to boil for forty-five minutes, and a further 5 gm., heating finally for another forty-five minutes. Allow the mass to cool, and very carefully add a little water. Rinse the whole out into a copper flask (glass vessels are useless), adding a solution of sodium hydrate containing about 25 gm. of the solid. Distil into a solution of normal sulphuric acid; about 15 c.c. diluted is usually sufficient. Each 1 c.c. of acid neutralised represents ·014 gm. of the nitrogen. It is advisable to make a blank determination on the materials used.

A modification of the above which can be recommended is to use 10 gm. of potassium sulphate instead of the manganese dioxide. In each case the heating must be continued until a clear, colourless liquid is obtained, and for at least one hour afterwards. If the clearing takes an unduly long time, it is most probable that the original material has not been sufficiently finely divided, and an agate mortar should be used. Coke is usually more difficult to decompose than coal. Pear-shaped flasks with long necks are especially suitable for the decomposition.

Coking Power of Coal.—This may be determined by heating a portion with varying amounts of sand. The more sand a coal can be mixed with, and still retain its coking power, the better its coking quality. The number of grams of sand per gram of coal forms a standard of comparison. At the bottom of such a scale we should find the non-caking coals, such as anthracite, while a strong caking coal would give as high a figure as 14 or 15.

An improvement on the above method has been devised by Dr Dunn.[1] He substitutes crushed anthracite for the sand in Campredon's test, passing the coal to be tested through a half-millimetre sieve, whilst the anthracite is powdered so as to pass through a millimetre mesh and be retained by a half-millimetre mesh. The various mixtures are coked in covered crucibles over a burner, but the writer prefers to use, say, five mixtures of 1-4, 1-8, 1-12, 1-16, 1-20, placing 3 gm. of each mixture in porcelain crucibles, each 1 in. diameter, and packing the five crucibles in one large porcelain evaporating dish with charcoal. The evaporating dish is then placed in a muffle at full red heat for half an hour, withdrawn, cooled, and the residues examined. With the proportions given above the writer has obtained quite distinctive results. The mixture, which turns out coherent, but can be crushed to powder by very slight pressure, is taken as the measure of the "agglutinating power."

The determination of the amount of soluble chlorides (common salt, etc.) in coal and coke may be effected as follows:—

From 10 to 20 gm. of a representative sample of the material is crushed to a fine state of division (passed at least through an 80 sieve), and boiled for an hour with from 100 to 200 c.c. of distilled water. This should be done under a reflux condenser, or the water may be renewed from time to time to compensate for evaporation. The liquid is then filtered, and the residue washed carefully several times with hot water. If desirable, that is, if considerable quantities of chlorides are anticipated, the filtrate and washings may be made up to a definite volume, and an aliquot part used for the titration. If preferable, the estimation may be done on the total filtrate. The solution must be neutral, or preferably faintly alkaline, which can be effected by the careful addition of dilute solution of sodium carbonate.

The process of estimation depends upon the fact that when silver nitrate is added to a solution containing a chloride and a chromate, the whole of the former is precipitated first before the chromate is thrown down. Further, the chloride is white, whilst the chromate of silver is red. The chromate, therefore, serves as an indicator of the exact moment when all the chloride has been thrown out of solution. Therefore, by using a standard solution of silver nitrate, an estimate of the amount of chloride present is easily calculated. By dissolving 23·94 gm. of crystallised silver nitrate in distilled water, and making up the solution to 1 litre (1,000 c.c.), a standard is obtained, 100 c.c. of which are equivalent to 0·5 gm. of chlorine, *i.e.*, 1 c.c. = ·005 gm. Cl.

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[1] "The Agglutinating Power of Coals." J. T. Dunn, D.Sc., F.I.C., *Jour. Soc. Chem. Ind.*, April 30, 1913.

By means of a burette, graduated in $\frac{1}{10}$ c.c., the above solution of silver nitrate is run into the solution containing chlorides, to which sufficient potassium chromate solution (free from chlorides) has been added to form a faint yellow colour. At first a milkiness is produced in the liquid, due to the precipitation of the white silver chloride. The solution is kept well stirred with a glass rod until the red chromate of silver which begins to appear just becomes permanent thus denoting the complete precipitation of the chloride For example, assuming that 20 gm. of coal are taken, and that the total solution obtained as described requires 4 c.c. of standard silver nitrate to precipitate the chlorides:

Then ·005 × 4 = ·020 gm. Cl in 20 gm coal, etc.
= ·100 gm. Cl in 100 gm. coal, or
0·100 per cent. chlorine.

This can be calculated into sodium chloride, knowing that 35·5 parts by weight of chlorine are equivalent to 58·5 parts by weight of sodium chloride.

This final volumetric determination can be so quickly and accurately made that the gravimetric method is seldom used. If it is desirable, the chloride present in solution can be precipitated as silver chloride by the addition of a slight excess of silver nitrate solution.

The silver chloride comes down as a white curdy-looking precipitate, which gradually turns to a bluish grey in presence of daylight. When settled, the precipitate is filtered off and washed with hot water several times, then carefully dried, heated to a low red heat, and weighed. This must not be done in a platinum vessel.

In calculating the result every 143·5 parts by weight of silver chloride are equivalent to 58·5 parts by weight of sodium chloride. The total chlorides are estimated finally by either of the above methods, but in order to get the whole of the chlorine a different initial treatment is necessary.

From 2 to 5 gm. of the finely ground material is mixed intimately with 10 to 15 gm. of lime, as free as possible from chlorides. It is an advantage, after mixing well, to add sufficient distilled water to make a stiff paste when stirred with a glass rod. The mass is slowly dried at a sufficiently low temperature to avoid "spitting" and consequent loss. It is now roasted at a low red heat at first, then somewhat hotter, until all the carbonaceous matter is destroyed. Excessive heat must be avoided, as it will cause volatilisation of chlorides. When cool, dissolve carefully in dilute nitric acid (about half and half) free from chlorides, add a moderate excess of silver nitrate solution, heat to boiling, allow to settle, filter off the precipitated silver chloride, wash thoroughly with hot water, dry and ignite, finally weighing the silver chloride. Calculate as previously described. An alternative method, and for several reasons preferable, is to boil the mass which remains after burning off the carbonaceous matter, with distilled water, which dissolves out the chlorides. Filter off from the insoluble lime, etc., and determine the chlorides in the filtrate either volumetrically or gravimetrically as previously described. In all these cases it is most important to make a blank determination on the whole of the materials used—minus the coal or coke.

Though the proximate analysis of coals is usually the one adopted for practical purposes, showing as it does the amount of ash, volatile hydrocarbons, fixed carbon, coke yield, and sulphur, it is occasionally desirable to make an ultimate analysis. Such a test, giving the total carbon, hydrogen, and oxygen, in addition to the results obtained in a proximate analysis, enables one to classify the fuel under certain heads, though commercial considerations must also be taken into account in such classification, such as coke yield, volume of gas, amount of tar and ammonia, the three latter of which can only be satisfactorily determined by a test made on a manufacturing scale, or under circumstances as nearly as possible approaching practical working conditions. In order to make an ultimate analysis, careful preparation and special apparatus are required, otherwise the results may easily be utterly misleading. A certain amount of experience in chemical manipulation is also necessary. The apparatus (Fig. 16) may be described as follows:—A porcelain or hard glass combustion tube K is supported on a combustion furnace, which is supplied with Bunsen burners suitably arranged for heating the tube and causing combustion of the coal or other fuel under examination. To each end of this tube is fitted a sound cork, bored and well fitted with glass tube through the centres. The fuel, which is being analysed, is mixed with copper oxide and heated, gently at first, then more strongly. A current of air or oxygen is passed through the tube, either under pressure at the end H, or by an aspirator, which draws air or oxygen through the apparatus from the other end C. The incoming air or oxygen must be purified by passing through washing towers P and Q. The first contains potassium hydrate to remove carbon dioxide, the other contains pumice stone soaked with strong sulphuric acid, which will retain any moisture. The purified air or oxygen, assisted by the cupric oxide with which the fuel has been mixed, ensures complete combustion of the fuel, the carbon being converted to carbon dioxide and the hydrogen to water. Any combustible sulphur compounds are at the same time converted to sulphur dioxide. Some lead chromate or lead peroxide must be packed loosely in the combustion tube in order to retain this sulphur, otherwise it will be absorbed finally in the potassium hydrate tube along with the carbon

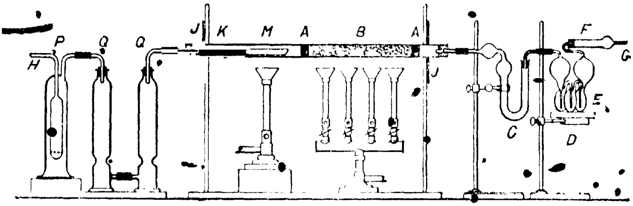


FIG. 16.—Ultimate Analysis of Coal, etc.

dioxide, and give too high a result for the carbon. The products of combustion are weighed after absorption in tubes C and E, which contain freshly dried calcium chloride and strong solution of potassium hydrate respectively. The calcium chloride must be free from lime (calcium oxide), otherwise some of the carbon dioxide would be retained in C, giving too high a result for the hydrogen and too low for the carbon. The gain in weight of tube C is due to water produced from the hydrogen of the fuel, and water contains 11·1 per cent. of hydrogen. The gain in weight of tube E is due to carbon dioxide from the carbon of the fuel, and carbon dioxide contains 27·2 per cent. of carbon. The object of mixing the coal with copper oxide is to prevent the carbon being incompletely oxidised, and forming carbonic oxide instead of carbon dioxide. The former would not be absorbed by the potassium hydrate in tube E, and consequently too low a result would be obtained. The routine of the analysis may be summarised as follows:—

1. Weigh tubes C and E very carefully.
2. Heat tube to dull redness, passing a current of pure air or oxygen through it, C and E unattached.
3. Allow end K of tube to cool.
4. Fit on tubes C and E.
5. Insert porcelain boat M with ·15 to ·2 gm. of fuel previously dried at 100° C.
6. Couple up tubes P and Q.
7. Pass a slow current of air or oxygen through the apparatus, and light the gas under the combustion tube; gradually heat to a dull red.
8. Gradually increase heat, taking care not to fuse the lead chromate.
9. Allow the process to proceed for half an hour at least.
10. Disconnect tubes C and E (closing their ends with rubber tube and glass rod), and weigh when cool.

The oxygen is estimated by difference. Sometimes oxygen and nitrogen are taken together, but this is unsatisfactory, quite reliable methods being available for the estimation of the latter. The amount of oxygen varies considerably, and is of great importance in the selection of coals for coking purposes.

CHAPTER VI

CALORIFIC POWER

THE calorific power or heating value of a fuel, and hence its evaporative power, can be determined by several methods, in broad principles the same, namely, the combustion of the fuel in oxygen (supplied either in the solid or gaseous form), and the measurement of the heat thus produced. It is possible also to calculate the calorific power from an elementary analysis of the fuel. The results, however, do not agree very closely with those found in a practical test.

In the Lewis Thompson calorimeter (Fig. 17) a weighed portion of fuel is taken and mixed with a suitable proportion of a mixture of potassium chlorate and potassium nitrate (three parts chlorate to one of nitrate), this supplying the oxygen necessary for the combustion of the fuel. After thoroughly mixing, the mass is transferred to the small copper cylinder A, and a fuse inserted. (The fuse is made from cotton wick strands, soaked in potassium nitrate solution, and dried.) The copper cylinder fits on a base B, on which three spring clips are fixed. Over all a copper cylinder C is secured by these clips, and a tube D, with a tap

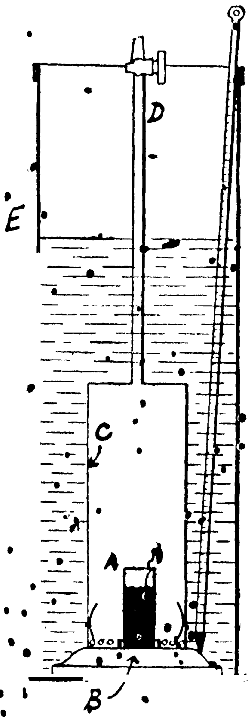


FIG. 17. Lewis Thompson Calorimeter.

attached, forms the outlet. The whole apparatus is contained in a glass cylinder E. In making a test a known quantity of water, usually 29,010 grains, is taken, and the temperature carefully noted, by a thermometer graduated in tenths of a degree Fahrenheit. Thirty grains of the fuel mixed with ten to twelve times its weight of the oxidising mixture are placed in the smaller cylinder, which is fixed in position. The fuse is inserted, lighted, and the outer cylinder immediately placed in position and secured by the clips. The whole apparatus is immersed in the water before ignition takes place, the tap being closed. The products of combustion pass through the small holes at the bottom of the cylinder, and give up their heat to the water. When all evidence of action ceases, the tap is opened and water allowed to enter the copper vessel, taking up the heat from the inside portions of the apparatus. The whole is shaken up thoroughly, and the temperature of the water again carefully noted. The apparatus itself takes up a certain amount of heat, and usually a correction of 10 per cent. is made for this.

Example :—

Weight of fuel taken	30 grains
Weight of water taken	29,010 „
Temperature before ignition	60·5° F.
Temperature after ignition	73·8° „
Rise in temperature	13·3° „

Then :—

30 gr. fuel have raised the temperature of 29,010 gr. water 13·3° F.

1 gr. fuel will raise the temperature of 29·010 ÷ 30 gr. water 13·3° F.

or 1 lb. fuel will raise the temperature of 967 lbs. water 13·3° F.

i.e., 1 lb. fuel gives off 967 × 13·3 British Thermal Units = 12,861·1 B.Th.U.

Allowing 10 per cent. for loss, we get a calorific power of 12,861 + 1,286 or 14,147 B.Th.U.

The figure 29,010 is used as being 967 times the weight of the fuel. By looking at above calculation it will be seen that the calorific power is 967 × rise in temperature. As it requires

967 heat units to convert 1 lb. water at 212° F. to steam, the number of lbs. of water evaporated (or converted into steam) will be, $\frac{967 \times \text{rise in temperature}}{967}$, or the rise in temperature plus 10 per cent. will give at once the evaporative power of the fuel. Thus in above case the evaporative power is 13·3 + 1·3 lbs. of water at 212° F. evaporated by 1 lb. of fuel, *i.e.*, 14·6.

In the William Thomson calorimeter, instead of using a solid oxidising agent, the gas oxygen is supplied either from a gas-holder or a cylinder. The combustion is started, as in the other case, by means of a short piece of fuse, or by a short platinum wire in an electrical circuit, which becomes hot enough to ignite the fuel when a current of electricity is passed through it. The coal or coke may be contained in a small platinum capsule, but in our experience this is not desirable, since the continued contact with hot carbonaceous matter renders the metal brittle in time. Small porcelain crucibles answer equally well; in fact we have often used the bowl of an ordinary clay pipe from which the stem had been cut off, loosely plugging the bottom with a layer of asbestos. The measurements of temperature and calculation of results are carried out as before. It is desirable to note the time occupied by the combustion, and afterwards take the fall in temperature experienced by the water during the same length of time. A correction for radiation is thus obtained.

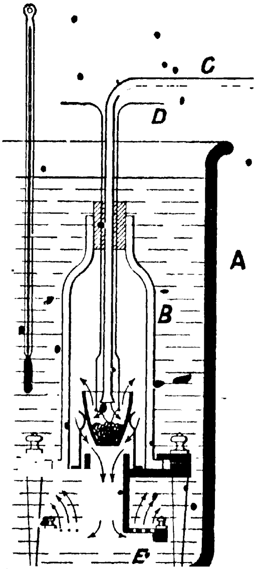


Fig. 18.—Thomson-Darling Calorimeter.

A modification of the Thomson type is shown in Fig. 18 (Thomson-Darling calorimeter). The combustion chamber consists of a glass bell jar B, which may be tightly clamped to a brass ring. Oxygen is fed into the combustion chamber by the tube C, and the fuel is ignited electrically by the wires D. The products of combustion pass into a flat circular distributing chamber E, as shown, the gases being split up into small streams by the perforated upper plate. This calorimeter has also been adapted for use with liquid fuels, a small brass lamp with asbestos wick replacing the crucible in this case.

The Bomb Calorimeter.—In this type of calorimeter, first designed by Berthelot, and improved by Mahler (Fig. 19), the combustion of the fuel is carried out in a strong metal vessel, made of steel or some special alloy, which is in some cases lined with enamel, in others plated with gold inside, to resist the corrosive action of the gases produced, and often nickel plated outside. Oxygen gas is supplied to this vessel, which is called the bomb or grenade, at a pressure of about twenty-five atmospheres. The fuel is placed in a small platinum crucible or capsule, over which is suspended a short piece of iron or steel wire. On the passage of an electric current through it this wire becomes white hot and burns; the drops of molten magnetic oxide falling into the fuel ignite the latter, and the combustion continues until all the fuel is consumed. In some cases platinum wire is used to commence the operation. The bomb has a tightly fitting lid, screwed on to a leaden washer. It is immersed in a vessel containing water, and the whole is contained in a case lined with non-conducting material to prevent any loss of heat by radiation. The heat of combustion is transferred through the bomb to the water surrounding it, the increase in temperature noted, and the calculation worked out on similar lines to those previously given, corrections peculiar to the instrument being allowed for.

Fig. 19 shows a modification of the Mahler bomb calorimeter designed by the late Mr Bryan Donkin.

By means of these various calorimeters it will be seen that the heat produced by the complete combustion of the fuel is measured. Generally speaking, however, the most satisfactory method of testing the quality of various coals is to make an evaporative test under actual working conditions. The burning qualities of the coal can then be noted also, as well as the kind and amount of clinker and ash which are produced. To make a test on these lines requires careful and special arrangements, and occupies much time. Hence the use of a calorimeter of some type is necessary, and there is no question that such instruments afford reliable comparative data.

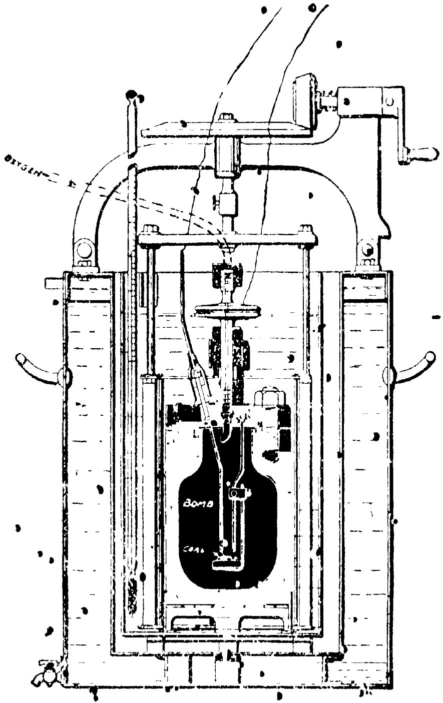


FIG. 19.—Mahler-Donkin Calorimeter.

An average calorific value for coal is about 7,500 calories, or 13,500 B.Th.U., that of coke somewhat less. It follows from this that 1 lb. of good coal should be able to generate 12 to 14 lbs. of steam. In actual practice, if 6 to 8 lbs. of steam are obtained per 1 lb. fuel, it is as much as can be expected. There are several factors which account for the difference between a calorimetric test and the practical results. Loss of heat by radiation; the impossibility of regulating the admission of air so as to supply exactly the amount required for the perfect combustion of the fuel. If too little, imperfect combustion occurs with production of smoke and carbonic oxide and consequent loss of heat; if too much air, then the excess uses up some of the heat in raising its own temperature to that of the other gases, which heat would otherwise be used for steam generation. Again, injudicious or careless firing leads to smoke and carbonic oxide production and loss of heat, and the capacity of gases for heat increases with their temperature.

It is possible to calculate the heating value of a fuel from its chemical composition, knowing the carbon, hydrogen, oxygen, nitrogen, sulphur, ash, and moisture. The substances which produce heat when a fuel burns are chiefly carbon and hydrogen, and sulphur to a slight extent. Any oxygen which may be present causes a loss of heat, because it uses up its equivalent amount of hydrogen, leaving only the excess as a heat producer. Moisture in the coal, together with that produced by the combustion of any oxygen present, takes up heat in its conversion into steam, and of course the mineral matter or ash is a non-producer of heat. Calculated heating values do not as a rule agree very closely with practical tests, or even with calorimetric results.

Two formulæ for calculating heat values are given:—

$$P = 8080\ C. + 34462\ (H - \frac{0}{8}) + 2250\ S.\ \text{(Dulong)}.$$

$$P = \frac{8150\ C. + A.\ \text{(volatile matter)}}{100}\ \text{(Gortal)}.$$

P = Calorific power in calories.

The former is based on the ultimate analysis, C. being the percentage of total carbon. The latter is based on the proximate analysis, C. = fixed carbon, *i.e.*, coke − ash. A. is a coefficient varying:—

13,000 between 2 and 15 per cent. volatile matter.
10,000 „ 15 and 30 „ „
9,500 „ 30 and 35 „ „
9,000 „ 35 and 40 „ „

The author's test on a coal of following analysis :—

C - - - - -	81·6	per cent.
H - - - - -	4·2	
O - - - - -	7·2	
N - - - - -	1·68	"
Ash - - -	4·0	
Volatile matter - - -	31·3	"
Sulphur - - - -	1·3	"

gave the following results :—

	Calories.	B.Th U.
Dulong formula - - - -	7,759	13,966
Goutal formula - - - -	8,246	14,843
Lewis Thompson calorimeter - -	8,030	14,454
Mahler bomb calorimeter - -	8,400	15,120

CHAPTER VII

HISTORY OF COKE MANUFACTURE

SOME coals are not suited for use in blast furnaces or foundry cupolas owing to their caking properties. The caking is due to the constituents of the volatile matter of the coal and the manner in which these constituents are grouped. The object of coke manufacture is to convert such caking coals into a fuel which is non-caking, richer in fixed carbon, and in better physical condition as to size, hardness, etc., for use in the blast furnace or foundry. In coking we aim at the complete expulsion of the volatile matter by means of heat, leaving the fixed carbon which, along with the ash or mineral matter, forms coke. In the early attempts at coking the coal was heated or coked in mounds or heaps in the open air (Fig. 20*a*), a portion of the heap being allowed to burn away completely to supply the necessary heat. In this case the volatile matter with its valuable by-products was allowed to pass into the open air, but a patent was secured by Jones in 1859 in which it was proposed to draw off the vapours from the old type of coke heap through a series of pipes, condensers, etc., as used in gas manufacture. The low yield of coke, however, condemned the open mound, and attempts were made to improve the process by confining the coal within walls (Fig. 20*b*), the necessary air for combustion of a portion of the charge being better controlled by means of dampers and flues, etc. In this case also the volatile matter was not made use of. The enclosed type of coke oven (Fig. 20*c*) then came into being, and the volatile matter, hitherto completely wasted, was burnt inside the chamber, supplying a great portion of the heat formerly obtained at the expense of the coke itself.

The shape of the enclosed type of oven was improved, and in the later beehive oven (Fig. 20*d*) is indicated by the name, the interior dimensions being usually about 12 ft. in diameter and

7 ft. high. The inner lining is of refractory brickwork, and the space between this lining and the outside walls is filled with sand, slag, or brickbats, to retain the heat. They are usually built in double rows, each oven being connected to a common flue, the connection being controlled by a damper. The waste heat from this flue passes through the boilers to the chimney. The operation of coking is carried on as follows: The doorway is partially built up, and the charge of slack fed into the oven from hoppers. Sometimes a small fire at the door is necessary to start the action, but when in full working order, the heat retained in the oven brickwork is usually sufficient. The charge is levelled and the door built up. For a few hours the gas comes off slowly, and is not of a quality to ignite, but after a short time the gas comes off more freely, and at this stage a little air is admitted above the charge to burn the gas, the heat from this combustion being reflected by the roof on to the charge below. The heat of the oven gradually increases, and the whole of the volatile matter is finally expelled. This requires about three days, and during the last portion of the coking the door is thoroughly luted to exclude air entirely, to avoid undue loss of coke. The yield of coke in these ovens, using slack containing 30 per cent. volatile matter, ranges from about 56 to 60 per cent., showing a loss of 10 to 14 per cent. through admission of air to the coking chamber.

When all trace of gas is absent the mass of incandescent coke is quenched inside the oven, involving a further waste of heat, and the charge is then raked out. To avoid this cooling of the oven itself by quenching, modifications of the oven have been built in which the charge may be drawn out by mechanical means and quenched outside; in some cases the waste heat flues are carried underneath the oven floor to supplement the heat from above the charge (Fig. 20*e*). Several attempts were made to recover by-products in ovens of the beehive type, and about 1870 extensive trials were made in this country by Messrs. Bell Bros. and The Wigan Coal and Iron Company, using the Pernolet type of oven (Fig. 20*f*). The type put down by the latter firm was a modification by Young, in which the gases were drawn off just above the coal by an exhauster, and after passing through a by-product plant, were fed into the furnace underneath the oven and burnt, the products of combustion thence passing through a casing enclosing the dome, and finally passing to the chimney. Aitken and Jameson also endeavoured to adapt the beehive-shaped oven for the recovery of by-products, both following the plan of downward suction. All these early adaptations failed on account of the doubtful quality of the coke produced. In the non-recovery beehive oven the tarry vapours had to pass upwards through the layers of hot coke, depositing a portion of their carbon by decomposition, and helping to cement the whole, giving finally the hard, silvery beehive coke. In the Pernolet, Aitken, and Jameson ovens the vapours were drawn away as fast as produced through the cooler layers, and no deposition of carbon took place. The portions of the charge farthest removed from the source of heat were distilled very slowly, and by the time the heat reached them the coking properties would be considerably lessened. The Pernolet oven was rightly designed in that the chamber was kept enclosed and air excluded as much as possible, but the distillation was after all a low temperature one, and consequently the tars produced were thin, containing a large percentage of paraffin bodies, whilst the sulphate yield was low. However, a later development of the Pernolet oven was erected at the Shamrock Pit, Westphalia, in 1886, in which a regenerative system was used. The charges were coked in comparatively shallow layers, and the quality of the coke was satisfactory. For truly coking coals the beehive oven is well adapted, and the beehive coke of this country and of the Connellsville region in the United States has deservedly a world-wide reputation, but on the Continent a great proportion of the coal merges into the lean variety, and it was found that to obtain satisfactory coke more rapid coking with higher temperature was necessary, and the Belgian type of oven (Fig. 20*g*) was designed for such conditions. Here we see the origin of the retort-shaped oven which, with narrower chambers, was utilised in the earlier Coppée oven. These proved so successful on the Continent as to merit serious consideration in this country, and in 1873 a battery of Coppée ovens was in operation near Sheffield. Many of these ovens were erected, but they have now been largely replaced by the modern Coppée and other recent by-product ovens.

The early Coppée oven (Fig. 20*h*) consisted of long rectangular chambers about 30 ft. long and 3 ft. 6 in. high. They were usually built with a slight taper, being 15 to 17 in. wide at one end, and 18 to 20 in. at the other, to lessen the friction of the coke on the oven walls whilst discharging. The ovens were charged from the top, and the gases from the ovens passed as shown into a series of vertical flues in which was also admitted the air necessary for combustion. This air was in some cases heated to a temperature of 700° F., by being conducted through passages in the hot brick-work underneath the ovens. The heated products of combustion passed downwards into a sole flue, and then, traversing the whole length of the oven, returned by the sole flue of the adjoining oven to the chimney flue, passing under boilers before reaching the atmosphere. The advantages gained in this oven were: Increased yield through exclusion of air from the coking chamber, shorter coking period, external quenching of coke, saving of time and labour through the use of mechanical appliances, etc. This oven has, of course, been considerably modified, and is now adapted for the recovery of by-products. The modern by-product oven may be looked on as resulting from the combination of the enclosed beehive chamber with external heating (Pernolet type), and the retort-shaped oven (Coppée type). The first attempts at this combination were made about 1869 in the Knab oven modified by Carves, and in 1881 the Carves oven, further improved by Simon, was introduced into this country.

Shortly after this the Semet-Solvay oven was brought out, but for some time the by-product oven made little progress, owing to the prejudice of the ironmasters against the appearance of the coke; but once this prejudice was overcome, the by-product oven made substantial progress, as indicated by the diagram in the first chapter. The development of coking systems is shown in Fig. 20.

(*a*) Coke heap, on the lines of the old charcoal pile.
(*b*) Coking between walls, air being more under control
(*c*) Approximate shape of earlier enclosed ovens.
(*d*) Modern beehive coke oven.

(*e*) Beehive oven with sole flue.

(*f*) Beehive oven adapted for extraction of by-products.

(*g*) Belgian oven designed for lean coals.

(*h*) Earlier Coppée oven. Narrow chambers heated from he sides.

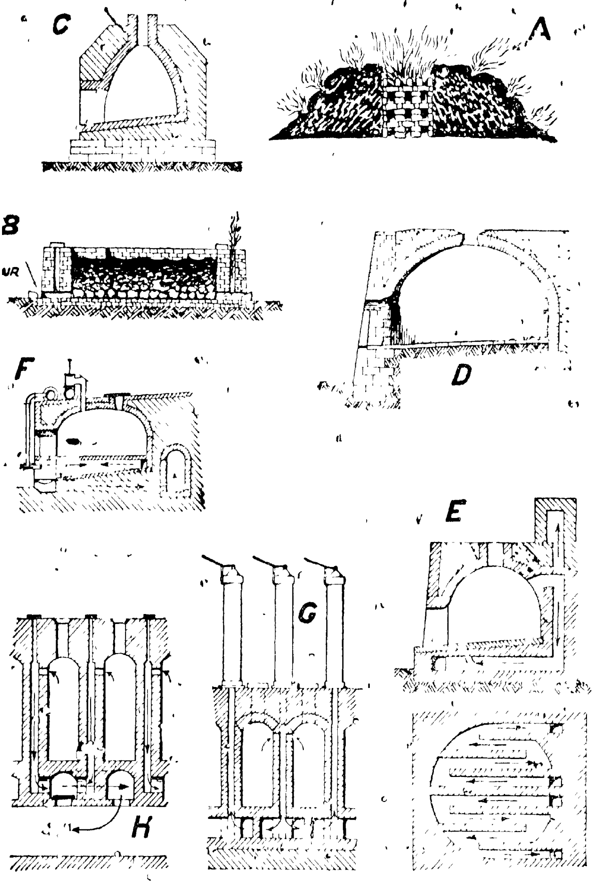


FIG. 20.—Development of Coking Systems.

CHAPTER VIII

DEVELOPMENT IN COKE OVEN DESIGN.

THE recent developments in coke oven design may be classified in various ways First, taking the arrangement of the coking chamber, this may be:—

(*a*) Horizontal.
(*b*) Inclined.
(*c*) Vertical.

(*a*) All modern coke ovens, with very few exceptions, have horizontal chambers.

(*b*) Ovens with inclined chambers have been primarily designed for gas manufacture, and scarcely come within the scope of this work. So far, although in existence in a few towns in Germany, only one example exists in this country, at Darwen, where a battery of inclined ovens has been erected, but of which no complete results have as yet been made public. The tendency in modern coking practice is in the direction of larger chambers, and in the author's opinion the erection of inclined chambers of large size must present considerable structural difficulties.

(*c*) These do not appear to have made headway. The type designed by A. O. Jones, in 1906 presented certain very desirable features, but many of these are also incorporated in the most recent type of horizontal ovens, in the way of greater depth of charge, cooler upper layers, etc.

Coke ovens may also be classified according to the direction of the heating flues, which may be:—

(*a*) Horizontal.
(*b*) Vertical.

(*a*) In the horizontal flued ovens the flues are usually capable of examination throughout their whole length, the regulation of heat being thereby simplified. On the other hand, the gases have a much longer travel to the chimney than obtains in a vertical flued oven.

(*b*) In the vertical flued ovens the gases have a shorter passage, and the gas stream is capable of being subdivided into many small sections of the flue system to assist in securing more uniform heating of the oven wall. The various designers of coke ovens have their own particular type, each with professed advantages, but the writer, after observation of several plants of both types, has not seen any marked advantage of the horizontal flue over the vertical flue, or vice versa.

A further very important classification relates to the method of utilising the heat in the spent products of combustion from the coke oven flues. According as the heat from these spent products is to any great extent returned to the oven flues or utilised for some external purpose, we may classify the oven as :—

(*a*) Regenerative.
(*b*) Waste heat.

The coke oven flues are heated by gas burnt with the correct proportion of air. In a regenerative oven the greater portion of the heat in the spent gases from the flues is transferred to the air supply. As this air supply is thus carrying considerable heat into the flues, a less supply of gas is necessary to maintain the desired temperature, and a greater proportion of gas is rendered disposable for purposes not directly connected with the coke plant itself, such as gas lighting, gas engines, etc. On the other hand, the transference of heat from the spent gases leaves them merely hot enough to create the necessary draught at the chimney, and no heat is left for any other purpose. The coke manufacturer cannot have it both ways. Of the heat units represented in the gas evolved from the ovens a surplus is available either in the form of "live gas" for lighting, power, etc., or "waste heat" for steam production, power, etc. His choice would be influenced largely by geographical conditions. If the plant were in close proximity to a town or well-populated district, a regenerative oven giving a greater proportion of spare gas would probably be a more attractive proposition. It may definitely be taken for granted that coke oven gas from a modern well-designed plant is quite suitable for lighting purposes, more especially under the conditions likely to obtain in the near future. As a matter of fact, the following towns in this country are already using very large quantities of coke oven gas:—Birmingham, Leeds, Middlesbrough.

In Germany, in the Dortmund area, in which the great majority of the coke ovens exist, the consumption of coke oven gas has advanced as follows:—

Year	Gas Sold Millions of Cub Ft
1908 - - - - - -	400
1909 - - - - - -	850
1910 - - - -	1,500
1911 - - - - -	3,000
1912 - - - - -	5,600

whilst the latest figures available from the United States indicate that over 120 million cub. ft. of coke oven gas per day are sold for illuminating and domestic fuel purposes.

The tendency in coke oven practice at the present time appears to lean towards the regenerative oven. At the same time a coke plant may be required for a district some distance away from industrial centres, possibly in the region of collieries requiring power for pumping, haulage, etc. Taking into consideration the high efficiency of a modern steam turbine, a waste heat plant in this case would be worthy of serious consideration. One point to be remembered in the case of waste heat ovens is the effect of a fluctuating load in the power house. A decrease in the load lessens the demand for steam, and since the waste heat cannot be stored, a portion of it at times must be by-passed to the chimney. In the case of a regenerative oven, the surplus gas may be conveniently stored in a holder during the slack periods to act as a reserve, thus avoiding waste of power.

A theoretical consideration of the principle of regeneration is useful. Taking one known example of waste heat oven in actual working practice, it was found that 72 per cent. of the gas evolved was burnt in the flues. This amounted to approximately 7,500 cub. ft. of gas of a calorific value of 485 B.Th.U. per cub. ft. (net) for every ton of coal coked. Thus each ton of coal required in round figures an admission of 3,600,000 B.Th.U. into the flues. For the particular variety and condition of coal dealt with in this instance, the figure remains the same whether cold air or pre-heated air is used, and the following calculation shows the effect of admitting air at increasingly higher temperatures into the coke oven flues.

	Analysis (Coke Oven).	Cub. Ft. Air per Cub. Ft. Gas.	B.Th.U. per Cub. Ft. (Net).	Weight. Lb. per Cub. Ft.	Weight of Consts. in 100 Cub. Ft.
	Per Cent				Lb.
Benzenes	1·0	35·8	3,574	·208	·208
Olefines	1·4	14·3	1,510	·078	·109
Marsh gas	28·8	9·54	919	·0447	1·287
Hydrogen	54·1	2·39	272	·0056	·303
Carbon monoxide	5·0	2·38	330	·0782	·391
Carbon dioxide	2·0		..	·1234	·247
Oxygen	0·4	..	..	·0893	·036
Nitrogen	7·3		..	·0784	·572
Average		4·72	485	·0315	3·153

Temperature of Air.		A. Heat Units in 5 Cub. Ft. Air (Less Units at Atmospheric Temperature)	B Heat Units. Gas + Air {485 + A} (Per Cub. Ft. Gas Burnt in Flues).	C. Cub. Ft. Gas (3,600,000 ÷ B) (Per Ton Coal Carbonised)	D. Surplus Gas (10,500 – C. etc.).
Fahr.	Cent.				Per Cent.
60°	15°	Nil.	485 B.Th.U.	7,422	29
572	300	50 B.Th.U.	535 ,,	6,729	36
1,112	600	106 ,,	591 ,,	6,091	42
1,652	900	164 ,,	649 ,,	5,547	47

In actual practice these results have been exceeded, and instances of 60 per cent. spare gas have been known. There are various methods of transferring heat from spent gases to the air supply. The simplest method is to lengthen the course of the hot spent gases, and to carry the air supply in the opposite direction through adjacent passages. The heat from the spent products is transmitted through the intervening wall to the air, but unless this wall is unduly thin, giving rise to a serious risk of leakage, or unless the passages are of great length, the efficiency of this method is low. A typical example is given in Fig. 21, an early recuperative system of Simon-Carves. A decided increase in efficiency is obtained in the Semet-Solvay continuous type of recuperator, in which specially designed blocks are used, and which is shown in Fig. 22. Though not quite as efficient as the reversing type, this has a decided advantage in that the

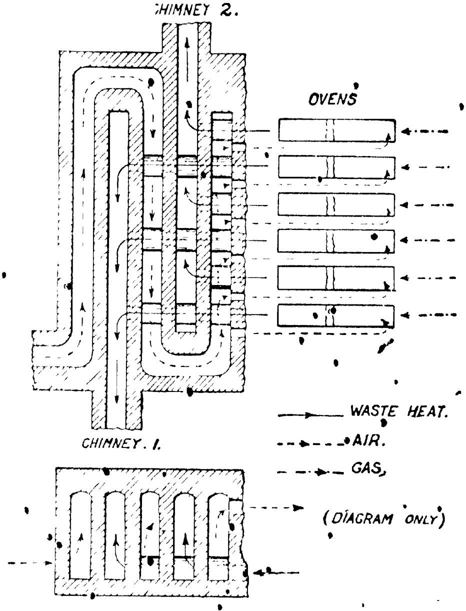


FIG. 21 —Early Recuperative System. (Simon-Carves.)

gas and air in the flues flow continuously in one direction, and the mechanical difficulties in reversing are avoided, as well as the alternations in the temperatures of the air. The earliest application of the Siemens principle of regeneration to coke oven practice was in the Otto-Hoffmann oven, the principle of which is shown in Fig. 23.

In this case the vertical flues are divided into two groups. The waste heat from the oven flues is being passed through

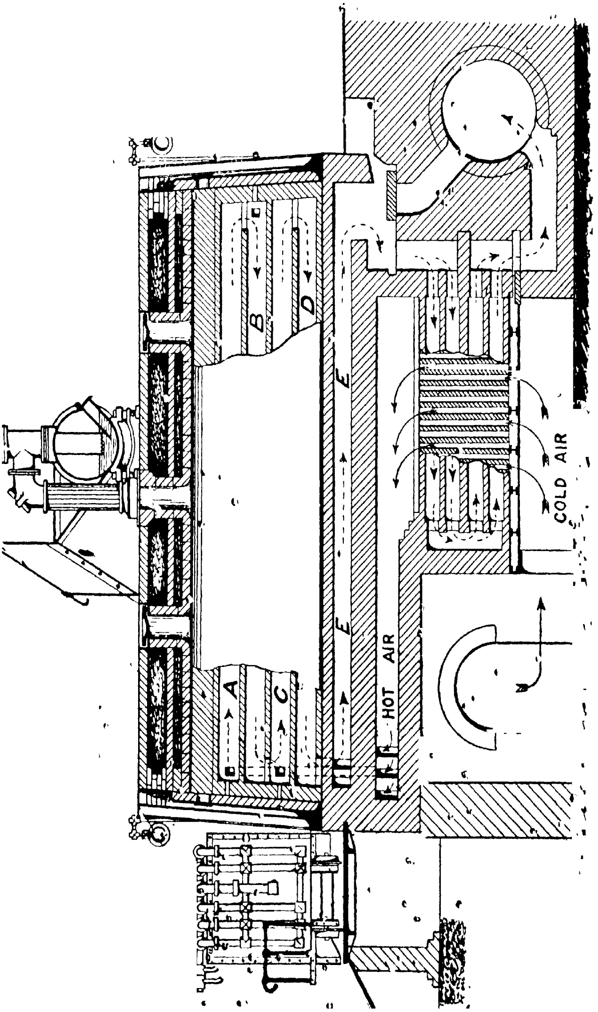


FIG. 22.—SEMET-SOLVAY CONTINUOUS RECUPERATOR.

the chamber A, filled with chequered brickwork, which in time becomes considerably heated. In the meantime cold air is being blown through the chamber B, which has been previously heated. The air becomes heated to a temperature between 900° and 1,000° C., and meets the gas in the combustion chamber C. The products of combustion rise in the left-hand section of the flues, and descend in the right-hand section. Thus, in the position shown in the diagram, the air and gas are travelling clockwise. This cannot go on indefinitely, otherwise the cold air would gradually cool B to atmospheric temperature, and the oven would become a waste heat oven. Consequently, about every half-hour the damper E is reversed to the position shown dotted, whilst all the gas cocks at G are simultaneously opened, and those at F closed. A little consideration of the diagram will show the new direction of air and gas, etc., to be now anti-clockwise.

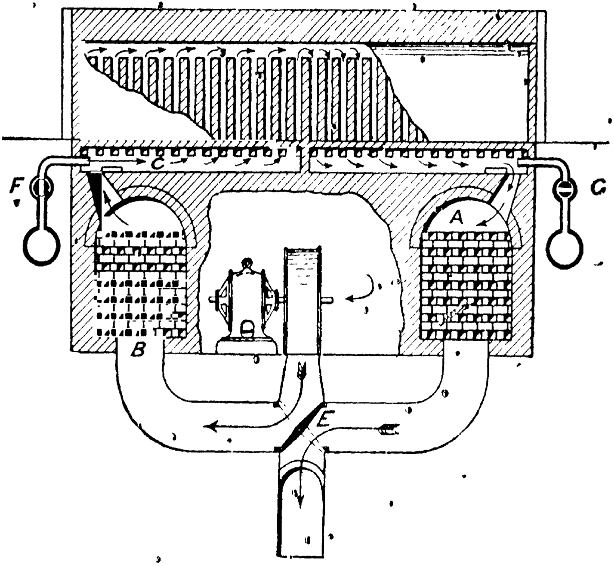


FIG. 23.—Reversing Principle of Regeneration.

The principle is the same in all types of regenerative ovens, variations occurring in the position of the regenerators. Some have regenerators common to all the ovens in a battery, others have separate individual systems of chambers for each oven.

A recent type of regenerative system, as designed by the writer, is shown in Fig 24. The essential feature consists of the use of two regenerators for each oven, each of these regenerators being divided into an upper and lower section to lengthen the path of the waste gases and air. The waste heat from any particular sole flue is deflected into the right-hand and left-hand chamber alternately, whilst the air passes through the remaining chamber. The dampers for this purpose are worked by cranks and connecting rods from a common shaft. If used as a reversing system, it will be seen that, whatever the position of the dampers, the regenerators for air and spent gases alternate. Thus air passing to the oven is always sandwiched between chambers receiving hot spent gases, and is always passing *in the opposite direction.* Thus the principle of a continuous system is incorporated in a reversing system. The lengthening of the courses of the products allows time for a portion of the heat to be transferred through the vertical dividing wall. The lengthened contact and counter-current system allows the air to pick up heat gradually, and avoids undue shock from admitting cold air direct into a hot regenerator, whilst the variation in temperature at the hot air distributing flue is lessened by utilising to a greater extent the principle of a continuous regenerator. All the reversing levers are on one side of the oven, and the dampers are arranged to permit of easy removal for repairs. A main reversing valve is not necessary, and the system avoids any reversal of gas cocks, the flow of gases in the flues being continuous. The position of the regenerative chambers relative to the oven battery allows individual repairs to any oven or regenerator, whilst the lower half of a regenerator, being the cooler, diminishes the heat losses by radiation into the foundations. The illustration shows the system adapted to a horizontal flued oven, but it is not intended that the system should be confined to this class of oven, it being possible in certain cases to adapt it to a vertical flued oven.

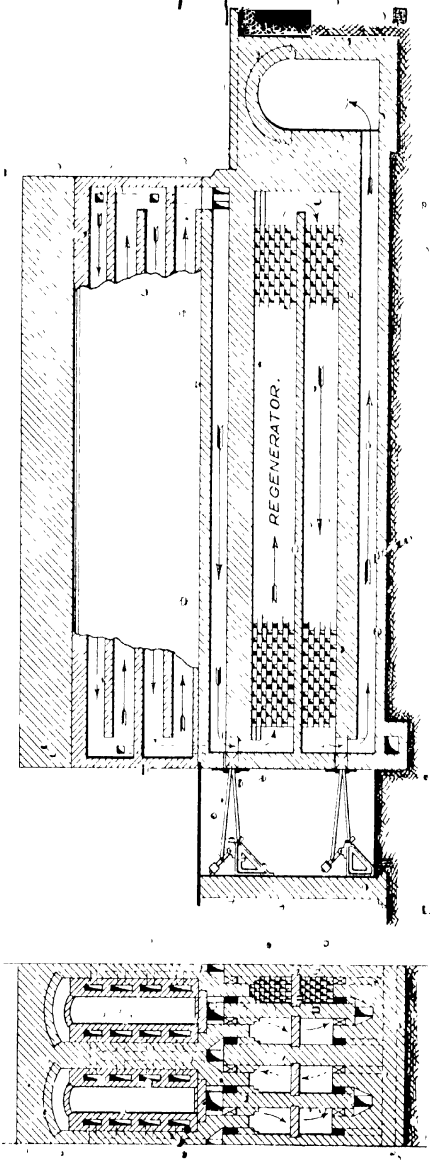


Fig. 24.—Combined System of Heat Recuperation (Christopher's Patent.)

CHAPTER IX

RECENT TYPES OF COKE OVENS

THE Semet-Solvay oven is a horizontal flued oven of strong, substantial design. In the standard type (Figs. 25 and 26) the heavy superstructure of the oven is supported by a division wall, the flues being entirely independent. This type has an undoubtedly long life and offers special facilities for repairs to the linings, a point of great value where coal of unduly high salt content is to be used The oven is strengthened by the brickwork of the division walls being of less refractory but tougher material than that of the flues themselves.

As well as materially strengthening the oven, this pillar acts as a reservoir of heat, a great advantage during temporary stoppages of the plant. The heat stored in the pillar also assists in counteracting the cooling of the flues through the introduction of a charge of wet slack. A somewhat cheaper type is also designed in which the flues are built into the division walls (see Fig. 26, B).

It will be noted that in the Semet-Solvay oven, each oven is entirely independent of its neighbour, having two series of side flues instead of a series of flues common to two adjoining ovens (as is the case in the majority of retort ovens). The heat of the oven is thus more readily controlled, and any single oven may be repaired without interfering with the working of the two adjoining ovens. To ensure gas tightness, a very important item indeed, the flues are usually built of small rectangular blocks about 8 x 4 x 2 in. In larger blocks the deformation or warping, to which all blocks are liable, is more accentuated, causing the joints to open and allowing a portion of the gas to pass into the side flues, thus adversely affecting the yield of by-products and surplus gas. These small bricks can readily be obtained with very uniform surfaces, and the jointing need not, with

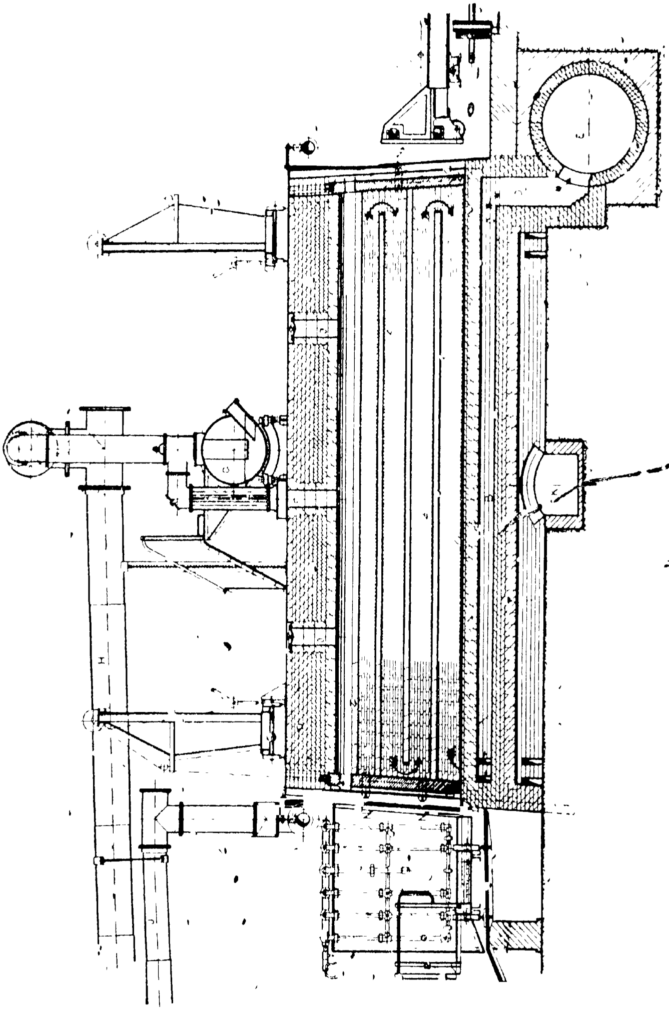


FIG. 25.—SEMET-SOLVAY OVEN.

ordinary care, be more than ⅛ in. in thickness. The gases from the oven, after being passed through the by-product plant, return to the distributing main from which they are fed into the flues as shown. In the waste heat type of Semet-Solvay oven, the air for combustion is preheated by being drawn through passages underneath the oven, to about 300° C. A type of continuous recuperator has been introduced, shown in Fig 22. It possesses the great advantage of being continuous in action, thus avoiding the sudden changes of temperature during the periodical reversing which takes place in the ordinary type of regenerator. At the same time the recuperator may be shut off for repairs quite easily without affecting the oven itself, using cold air in the meantime.

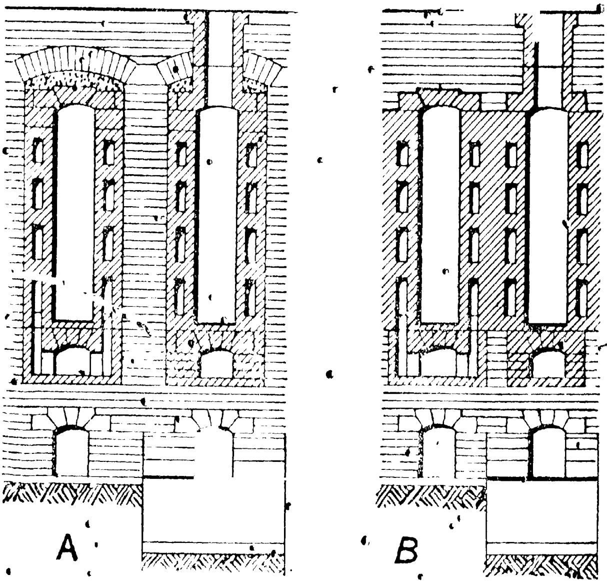


FIG. 26.—Sections of Semet-Solvay Ovens.

In the Semet-Solvay recuperative oven the air is preheated to temperatures up to 900° C. Gas and air are admitted into the top flue, the air in this flue being in excess, in order to prevent overheating of the top of the oven. The gas and air supplies are reinforced in the second and third flues to bring about the correct proportions of gas and air for efficient combustion, whilst in the fourth flue this

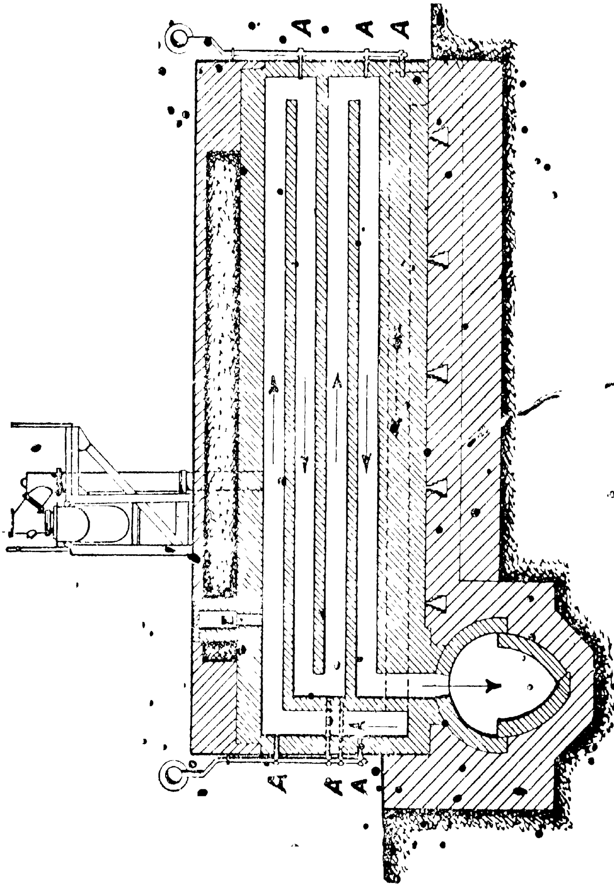


FIG. 27.—Huessener Oven. (Coal Distillation Co.)

combustion is completed. Typical temperatures of the flues are:—

Top flue, 950° C.; second flue, 1,050° C.; third flue, 1,100° C.

The Semet-Solvay oven is also built with reversing regenerators, the burnt products travelling downwards through the flues during one period, and upwards during the other. Each design is generally built with four flues, though sometimes five are used, but in the United States this oven is usually of greater dimensions, often six flues high and coking 16 tons per charge. The practice there is in favour of higher heats and consequently more rapid coking, charges being worked off in about eighteen hours.

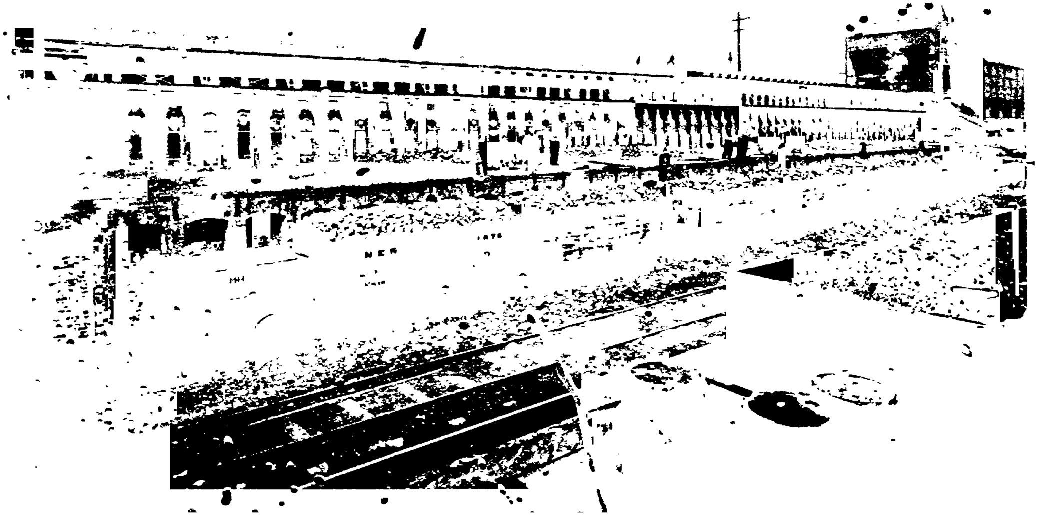
The Huessener oven (Figs. 27 and 28) is the type of waste heat oven adopted by the Coal Distillation Co., Middlesbrough, and is also of the horizontal flued type. The arrangement of the flues is somewhat similar to the original Simon Carves oven, but, like the Semet-Solvay type, each oven has its own side flues, and is entirely independent of the adjoining ovens. The flues are constructed of refractory tiles and bricks well dovetailed together. The air for combustion is not preheated. The gas and air supplied are well under control, and good heats are obtained in the flues, the following temperatures being given by C. Lowthian Bell in a valuable paper on the Huessener oven, read before the Iron and Steel Institute in 1904.

Bottom side flue, 1,900° F., 1,037° C.
Top „ „ 2,200° „ 1,205° „

The gas supply is well distributed, being admitted to the flues at twelve different points in each oven, six on each side of the ovens. The regenerative oven of the above firm is the Collin oven described later.

A somewhat similar type of oven is adopted by the Simon-Carves Coke Oven Construction Co., to meet the wishes of clients specially desiring a waste heat oven with horizontal flues. The main principle will readily be seen from the drawing, Fig. 29.

The air is not preheated, but is admitted through special



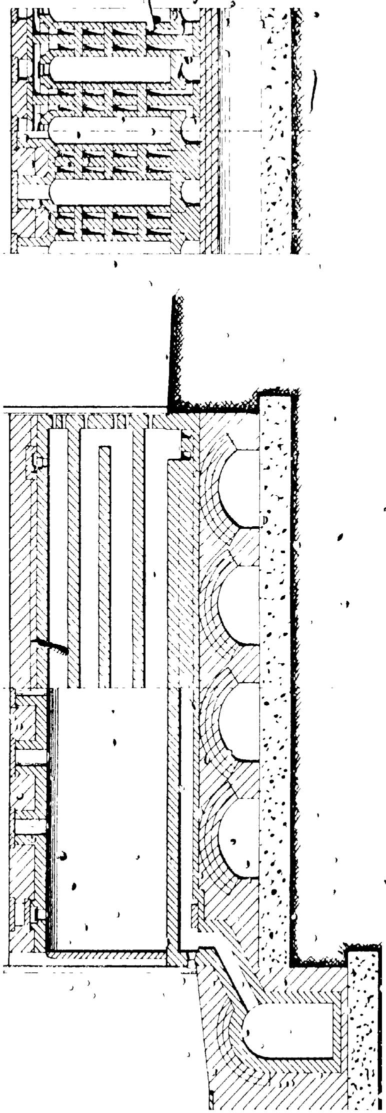
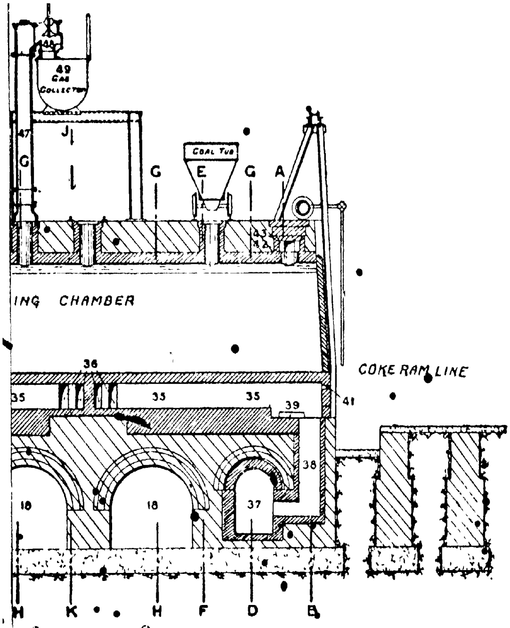


Fig. 28.—Simon-Carves Oven, Horizontal Flued Oven.

regulating devices incorporated in the armour plating supporting the buttresses. The same firm have also a vertical flued oven adapted as a waste heat or regenerative oven. The special features of both these types lie in the arrangements for controlling the gas and air supplies to the flues The waste heat type is shown in Fig. 30. The gas is fed into combustion chambers 11, 12, 13, and 14, by means of firebrick tubes and the gas cocks 3, 4, 5, and 6. The air is preheated in passages 18 in the oven foundations, and passing along 19 and 20 is subdivided by the tubes 25, 26, and 27, each tube possessing a special damper. These dampers are within easy reach of the operator. The gas cocks being also adjacent, the air and gas for each section can be adjusted to a nicety, whilst the subdivision of the flue system into eight sections assists in securing uniformity of heating along the oven wall. The regenerative type is shown in Fig. 31. In this type the system of admitting gas and air by specially designed firebrick tubes likewise obtains, but the oven wall is divided into ten sections instead of eight. The same facilities for control of gas and air are present in this type as shown in the drawings and emphasised in Fig. 32.

The Otto oven as now built is a distinct improvement on the earlier Otto-Hoffmann oven. The vertical flue has been retained, but the distribution of gas to the flues has been considerably modified, the special feature being the "underfired" system of heating the flues. In the waste heat type (Fig. 33) the gas is fed into the combustion chambers by sixteen Bunsen burners, the necessary air being drawn in, partly at the burners and partly through ports in the side of the combustion chambers. These burners are easily accessible, and the combustion of the gas is well under control, whilst the subdivision of the flue system into several distinct sections allows a uniform heat to be maintained throughout the length of the oven wall.

The gas ignites at the level of the coking chamber, and rises vertically through the heating flues, following its natural tendency. The chimney draught is thus decreased, and the loss of gas is reduced to a minimum. The regenerative type (Fig. 34) is also "underfired," but the air is preheated in the regenerators, specially arranged as shown, to allow easy access for inspection and repairs, etc.



SECTION P Q

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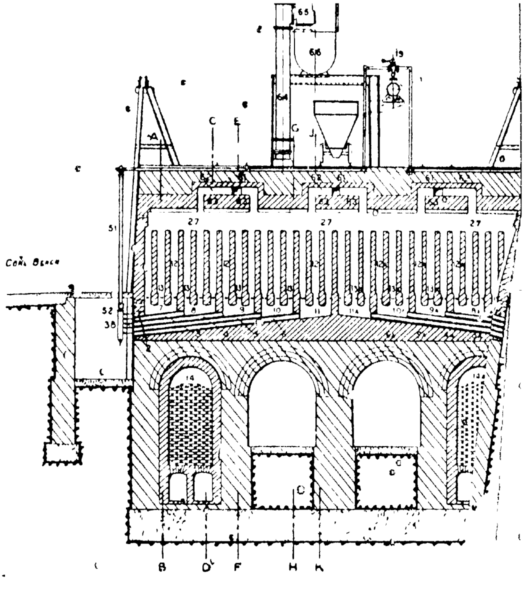
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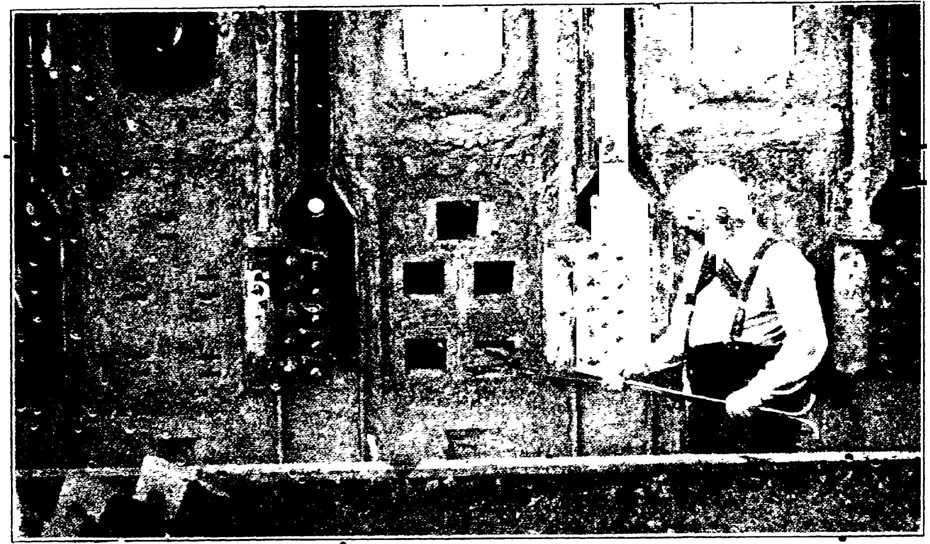

FIG. 32.—REGULATING FLUES, SIMON-CARVES OVENS.

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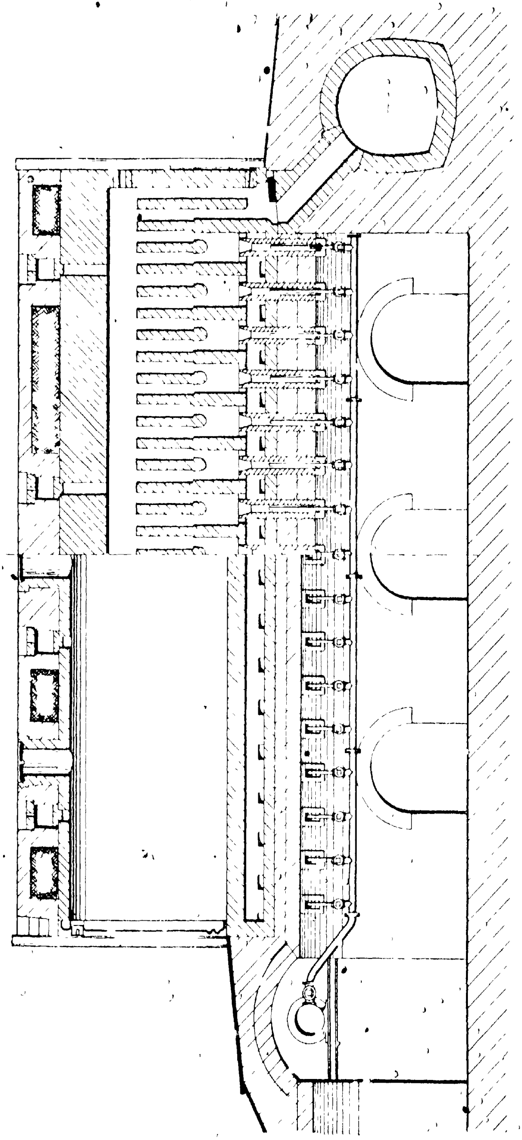

FIG. 33.—OTTO WASTE HEAT OVEN.

The Koppers coke oven is of vertical flued type, possessing many striking features in the heating arrangements of the flues. The oven is built in two types—waste heat and regenerative. The waste heat oven is of very simple construction. The gas, after being deprived of its by-products, is fed from the distributing main C into the gas distributing channel E formed of firebrick pipes. The gas passes from this channel through orifices, each fitted with a gas nozzle, into the vertical flues. These flues, numbering from thirty to thirty-five, have each a separate nozzle, the details of which are shown in Fig. 36. The nozzles have oval shaped orifices, and by means of a rod with a T end may be very easily changed. These nozzles have orifices of varying size, so that the amount of gas passing into each individual flue can be perfectly adjusted. The air necessary for combustion is drawn in by the chimney draught through the air distributing channel G from the air conduit F. From this channel it issues by the ports H into the combustion flue K, flowing round the nozzle U and keeping it comparatively cool. The amount of air is controlled by the unique damper arrangement shown in Fig. 36. As both dampers and gas nozzles may be adjusted from the top of the oven, the combustion of gas in the flues can be easily controlled so as to maintain an exceedingly high state of efficiency, and the greater number of gas jets ensures a uniform temperature of the oven walls from end to end. The products of combustion pass into the waste heat flue through a passage regulated by the damper R.

The regenerative type of Koppers coke oven is shown in Fig. 37. This is an improvement on the original Koppers regenerative oven, in that each oven has an entirely separate regenerator which allows of repairs to individual ovens without affecting the remainder of the battery. The reversing of the gas and air is done simultaneously along the whole of the battery by a link arrangement shown in Fig. 38. It is found that the waste heat is more than sufficient to maintain the heat in the regenerators, and consequently arrangements are provided for taking off a portion of the products of combustion through the gas flues T and V, Fig. 37. The arrangements for regulating the combustion in the individual flues are the same as described in the waste heat type.

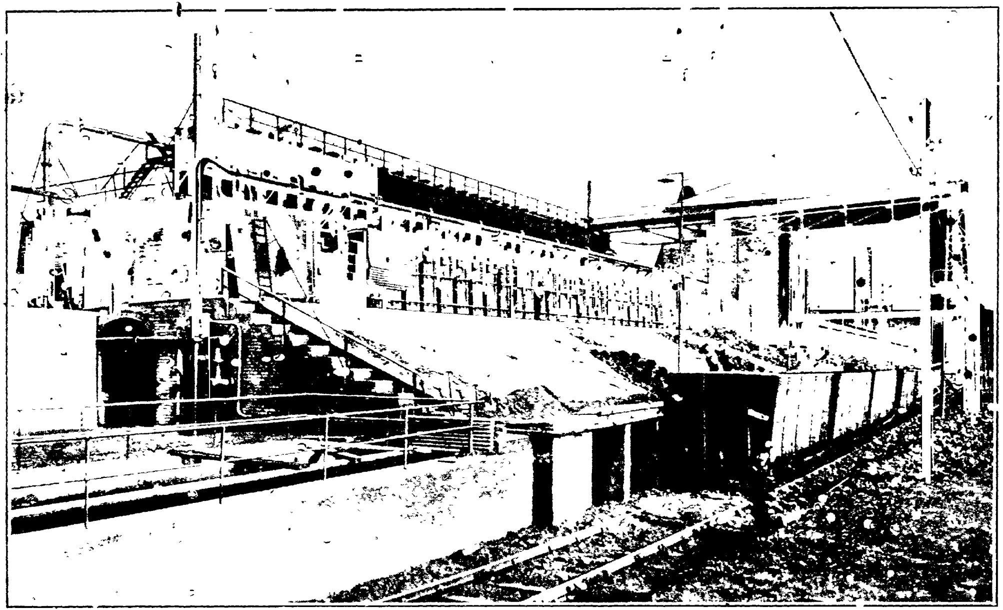

FIG. 35.—BATTERY OF OTTO OVENS.

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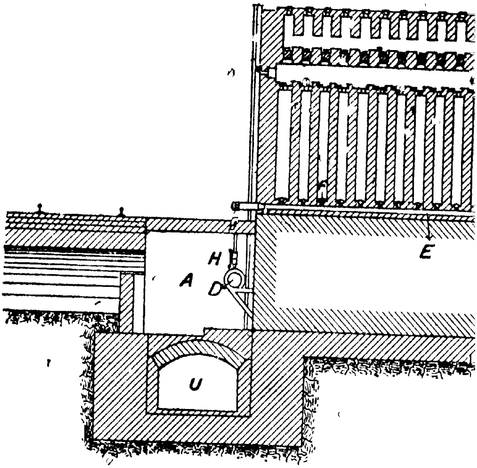
H
A
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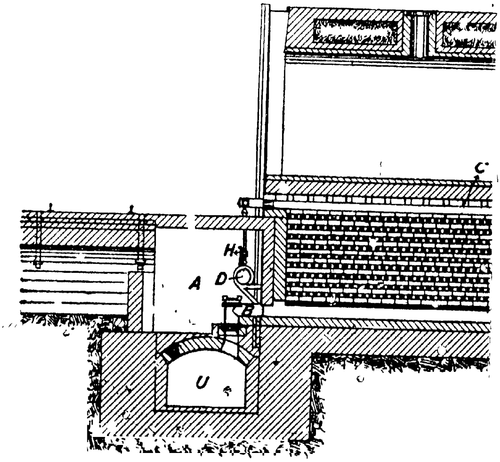
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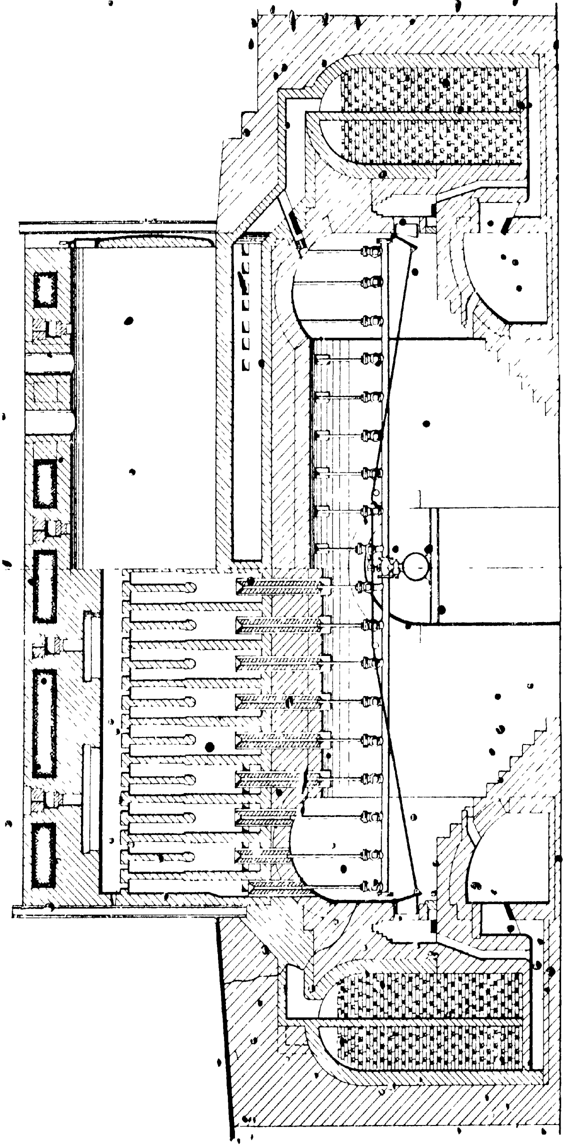


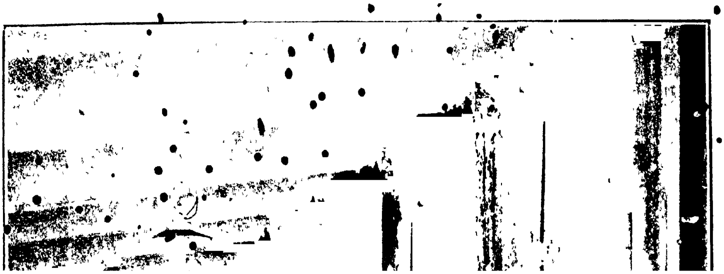
FIG. 34.—OTTO REGENERATIVE OVEN.

The Collin oven (Coal Distillation Co., Middlesbrough), Fig. 40, is of the vertical flued type with or without regenerators. The construction of this type of oven will be seen from the sections which show a series of flues with bond stones between. These bond stones are hollow, forming flues which, in the case of the waste heat oven, serve to lead the products of combustion to the main waste heat flue. In the case of the regenerative oven, these flues serve two purposes:—

1. To take off the waste gases after combustion in the main vertical flues.
2. To conduct hot air from the regenerators to the gas from the upper distributing channels during another period of the coking process.

The sectional area of these inner flues has been carefully predetermined to give the correct proportion of gas and air.

The principal feature of the Collin regenerative oven lies in its method of heating. This is carried out in alternative periods from the bottom and from the top. Gas is introduced at both ends of the oven from the distributing main B, passing along two series of conduits connected at the centre of the oven at E and N respectively, and during the period when the heating takes place from below the gases introduced at L issue through nozzles set at the bottom of flues K, the air at the same time coming from one of the regenerators through sole flues of one oven and the conduits, meeting the gas at the bottom of flue K. The products of combustion rise in K, and descend through the inner flues A in the bond stones and into the sole flue of the alternative oven, and thence to the regenerator. When the heating takes place from above, the gas is admitted to the upper portion of the oven walls at M but well below the level of the coal charge. It issues again through a number of nozzles into flues K, while the air for combustion rises through the inner flues of the bond stone A. The flame and products of combustion descend through flue K, and leave through conduit O and sole H into the regenerators. In this oven all the main vertical flues are at all periods heated directly by flames and not alternatively by flames and products of combustion, while alternative heating from bottom and top causes a very even distribution of the heat throughout the oven. At the same time the zone of



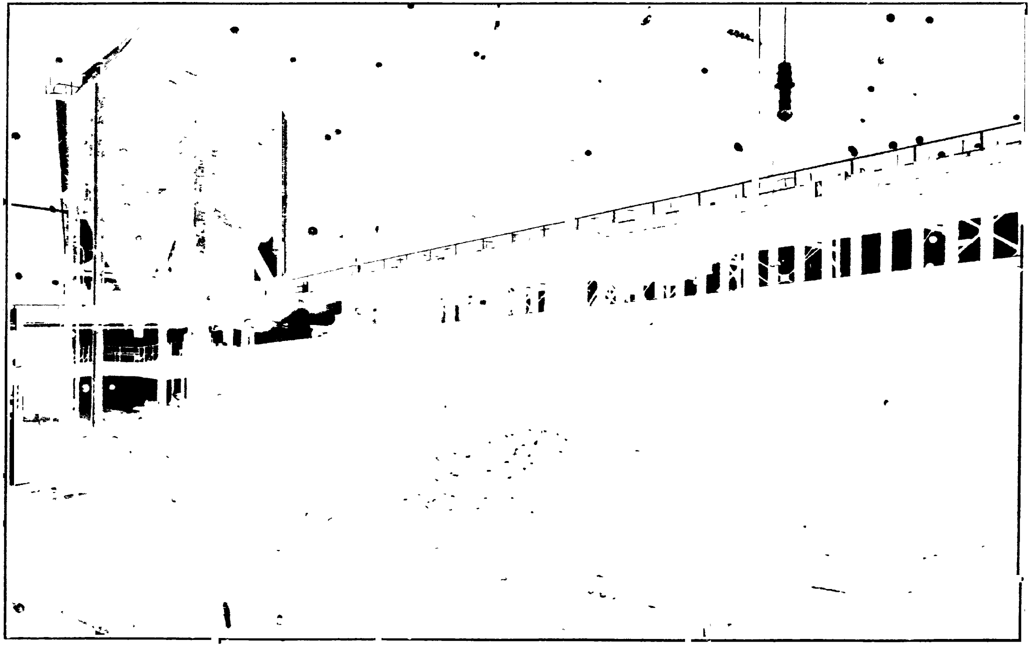


FIG. 39.—BATTERY OF KOPPERS OVENS.

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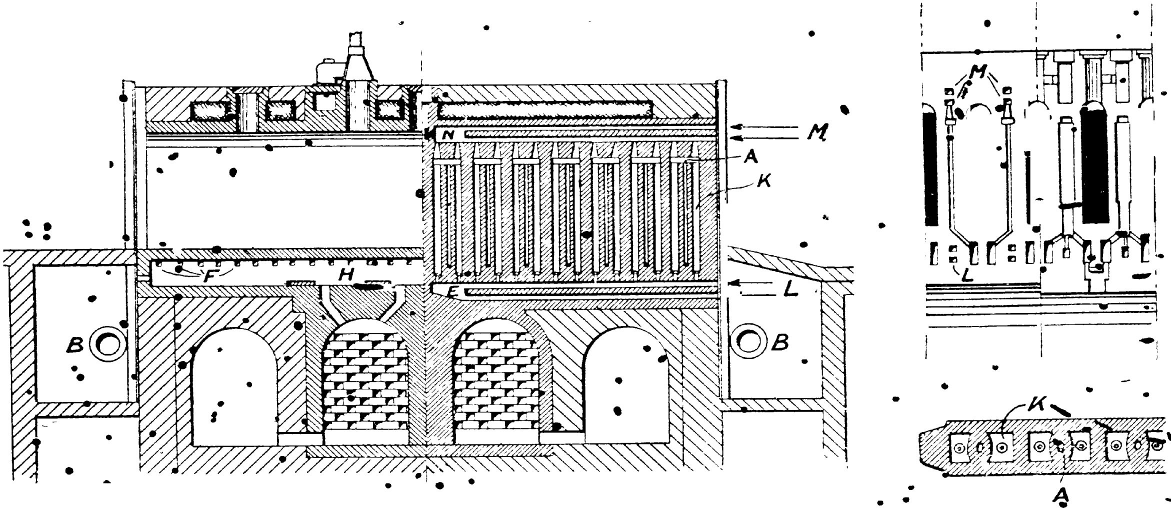


FIG 40 — COLLIN REGENERATIVE OVEN (Coal Distillation Co)

combustion for the upper admission of gas is kept so low as to not cause any overheating of the top portion of the oven. The reversing of the regenerators in some types of ovens causes an alternating variation of temperature from one half of the oven wall to the other, but in the Collin oven the heating is practically continuous.

The Coppée oven as now designed is the result of continued improvements on the original Coppée oven described on p. 53. The oven, batteries of which have been built at a large number of important collieries and steel works, is constructed for by-product recovery, and is a vertical flued oven designed on the waste heat or regenerative principle. In the waste heat type the heating wall is divided into two portions, each consisting of fifteen vertical flues. A tube of refractory material, fed by a Bunsen burner, delivers gas and primary air into the base of fourteen of the flues in each half of the wall. Secondary air to complete the combustion is also admitted, the amount being controlled by dampers, etc. The products of combustion unite in an upper horizontal flue, and from thence descend through the remaining flue of each half of the wall to the chimney flue and boilers, etc.

The regenerative type possesses special features, an important point being in the division of the heating flues into five sections, each section consisting of six flues, the periodical reversal taking place in each of these sections. The principle is shown diagrammatically (not to scale) in Figs. 41, 42, and 43, corresponding portions of the system being lettered alike in each case. By means of the gas cocks C controlled by levers L the gas is fed into flues 4, 5, and 6 in each section for one period, and into flues 1, 2, and 3 for the next period. The sole flues A and B serve to supply hot air (1,000° C.) to the flues, or to exhaust the spent products of combustion according to the position of the reversing damper D. In the position shown, the levers L are pulled by the chain NM in the direction of N, flues 4, 5, and 6 in alternate ovens thus receiving gas. Owing to the position of the damper D air is being blown through the regenerator $A_{//}$ to the distributing chamber $A_{/}$, connected to sole flues A which feed the vertical flues in each section as shown. At the same time the sole flue B by means of the suction of the chimney draught is

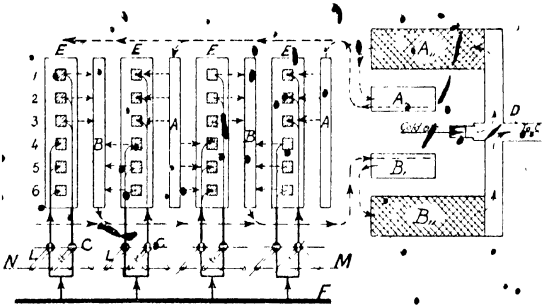


FIG. 41

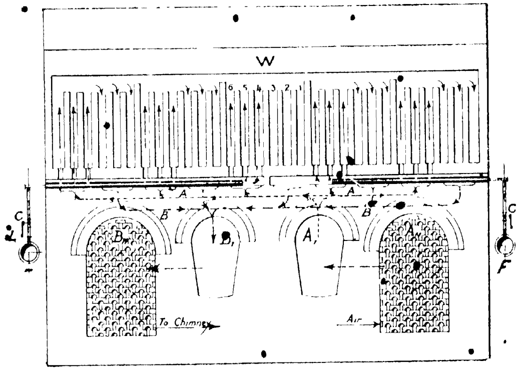


FIG. 42

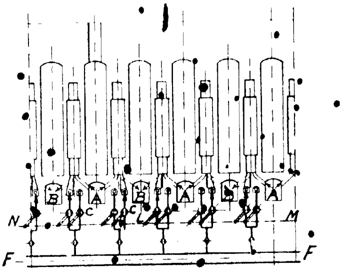


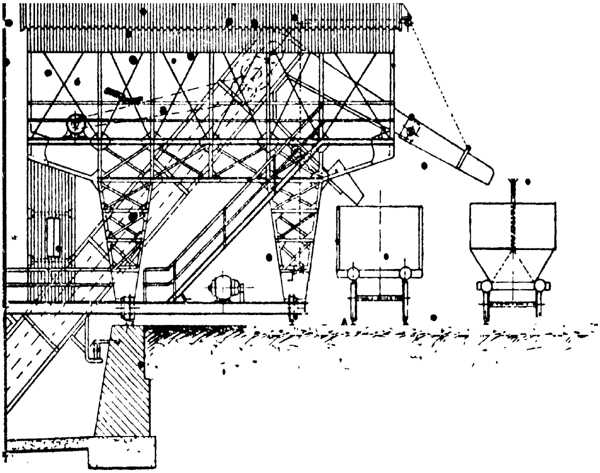
FIG. 43.

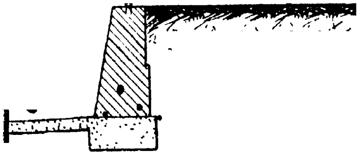
Coppée Oven (diagrammatic only).

collecting the spent products passing down the flues of each section. From B these heated products unite in the chamber B_1, and passing thence through the regenerators B_{11} give up their heat to the chequered brickwork in readiness for the next reversal. It must be noted that the reversal of the damper D and the cocks C is simultaneous. The chambers A_1 and B_1 are useful in causing the air to traverse the whole length of the regenerator, namely, from one end of the battery to the other, before being admitted to the flues. The reversal of the currents affects only three flues at a time or one-tenth of the oven wall. Thus the flues for the descending gases are in close proximity to, and nearly as hot as the ascension flues, and a uniform temperature is maintained throughout the length of the oven wall. A typical arrangement of this oven, with the necessary mechanical appliances, is shown in Fig. 44.

One of the most recent types of ovens is the "B.C.O." oven (British Coke Ovens Ltd.), a vertical flued oven, waste heat or regenerative type, Fig. 45. In many regenerative ovens two regenerators are used, but in the B.C.O. oven the special feature is the use of four regenerators, A, B, C, D, giving considerably greater cubical capacity. The regenerators are also individualised for each oven by cross division walls E, securing more equal expansion and contraction. The gas is admitted by means of two tubes F at each end of the oven, these gas ducts being kept cool by adjacent passages through which air is continuously circulating, thereby preventing decomposition of the gas and deposition of free carbon in the gas ducts. The two outer regenerators, A and D, serve for one oven, the two inner regenerators, B and C, serving for the adjacent oven and so on alternately. In the waste heat type the distribution of gas to the flues is on similar lines, but the air currents which keep the gas ducts cool are utilised to supply the air for combustion in the flues themselves.

Another recent type of regenerative oven, as erected by Bagley, Mills, & Co., 32 Victoria St, Westminster, is shown in Fig. 46. Gas is admitted from the main D into the right or left hand distributing chamber E according to the position of the main reversing dampers. The gas then rises through a series of nozzles F. Each unit F consists of a





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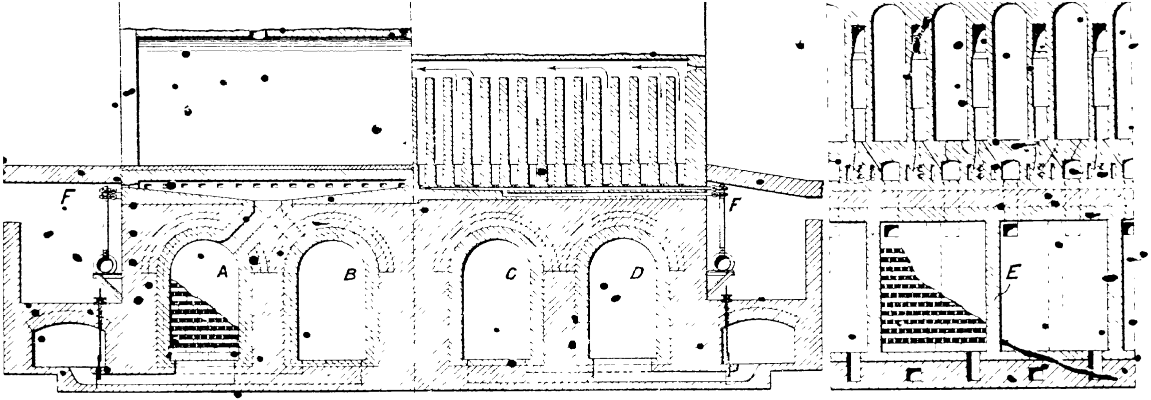
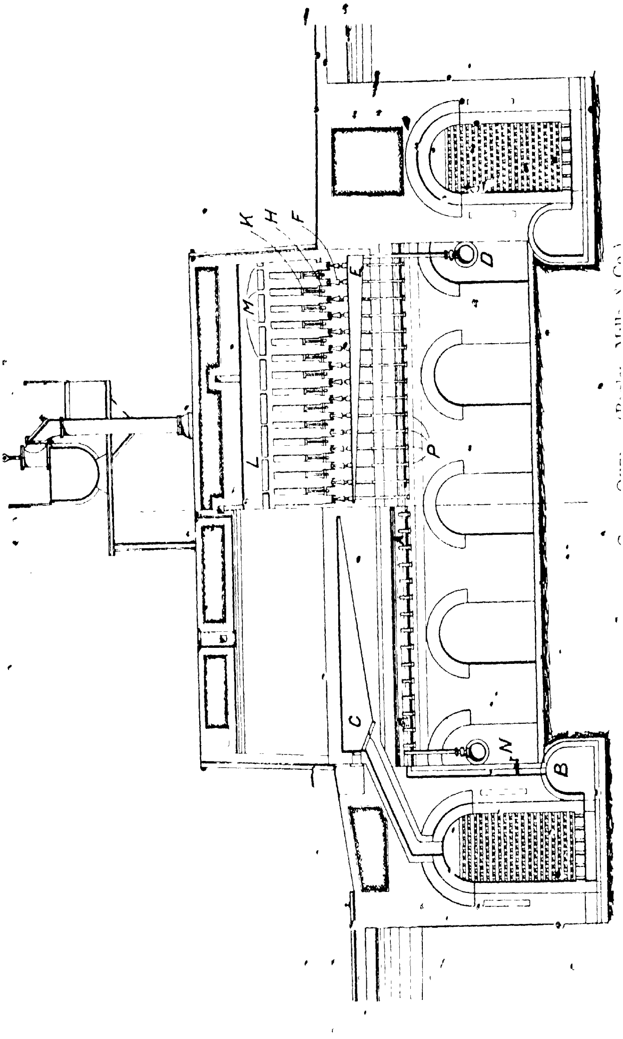


FIG. [illegible]5.—B.C.O. OVENS. (British Coke Ovens Ltd.)


K
H
F
M
E
L
P
D
C
N
B

lower nozzle designed to admit the exact quantity of gas for any particular flue, and an upper nozzle acting as a protection to the lower. The orifice in the upper nozzle is greater in dimension than the lower orifice, so that in the event of decomposition of the gas and deposition of carbon only the upper nozzle is affected and the supply of gas is always kept uniform. These nozzles may be examined at any time by means of mica capped tubes P. Air from a distributing chamber as at C passes to the centre of each flue partition, and from thence is admitted to the flues at two places, H and K. The openings are proportioned to allow of partial combustion at H and a completion of the combustion at K. By this means any tendency towards overheating in the lower section of the coal charge is neutralised and a more uniform heat obtained.

The products of combustion unite in the flue L, the natural tendency towards greater draught at the end nearer the chimney being corrected by carefully regulated openings M so as to ensure equal conditions for all the flues. It may also be noted that the air passing to the regenerators is carried at A, in close proximity to the heated under parts of the ovens, and then passes to the distributing chamber B, being controlled by valves N which are closed or opened simultaneously with the reversal of the gas. By this arrangement the incoming air acts as a cushion between the oven structure and the regulating galleries, thus securing more equable conditions in the latter Batteries of this type are in successful operation in Great Britain.

CHAPTER X

MECHANICAL APPLIANCES AT COKE OVENS

THE selection of the fuel for coke ovens is of great importance. The effect of impurities has already been discussed, but the nature and character of the residue left after closed distillation must also be considered. The range of fuels suitable for the manufacture of coke is indicated in Fig. 3. With the beehive coke oven, the range is extremely limited, and only such coals are suitable as are naturally of a strongly coking character (shown on the diagram as "coking coal"). With a modern retort oven and special treatment we can extend the range so as to include a proportion of lean coal, gas coal, and even flaming coal. There is still a fair amount of uncertainty as to what constitutes the coking power of coals. In the first place there must be sufficient volatile matter with its proportion of tarry compounds to bind the particles together into a uniform mass. On the other hand there must not be an excess of volatile matter. This is usually found in conjunction with a high oxygen content, and in the majority of cases as the oxygen increases the coking property is diminished.

G. S. Cooper,[1] in a paper read before the Iron and Steel Institute, gives the following results of investigations concerning the effect of oxygen content on the coking property of coal. He states that "it is generally assumed that a coal with more than 8 per cent. oxygen on an ash and moisture free basis is a non-coking or a poor coking coal."

Some of the results of tests are reproduced on the following page.

He arrives at a general conclusion that if the yield of water is more than 7 or 8 per cent., the coke is of an inferior character. The author's experience would place this figure higher, say from 9 to 10 per cent. With careful treatment, coal giving off 11 per cent. of watery liquid has been converted into satisfactory coke. If we examine the table below we find that the volatile matter in the coals giving good coke ranges from 20 to 32 per cent.; fair coke, from 32 to 35 per cent.; poor coke, over 35 per cent. A laboratory test on the volatile matter of a coal is, however, not satisfactory in drawing conclusions as to the coking power of a coal. It is difficult to secure conditions in any way approaching those

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[1] "By-product Coking in Relation to Iron and Steel Manufacture." G. S. Cooper, B.Sc., Iron and Steel Inst., 1914.

Coal.	Percentages.				Remarks.
	Coke.	H_2O.	CO_2	CO.	
Cumberland - -	69·8	5·31	0·94	6·0	Good coke
Durham - - -	76·5	4·11	1·02	4·6	,,
Warwick - - -	62·0	12·98	3·60	10·6	Poor coke
Monmouth - -	80·0	3·37	0·51	3·2	Good coke
Staffs. - - - -	71·0	7·53	1·08	4·9	,,
Yorkshire - - -	70·0	6·66	1·28	7·8	,,
Lancashire - - -	70·0	6·80	1·48	5·2	,,
Lanark - - -	65·0	8·09	2·45	10·7	Fair coke
Fife - - - - -	68·0	9·12	1·99	7·6	Good coke
Northumberland - -	67·0	8·55	2·46	7·8	Fair coke
Forest of Dean - -	68·0	6·73	1·58	7·5	,,
Brown coal, Australia -	18·0	12·78	8·22	23·0	Poor coke
Leicester - - -	63·0	10·52	2·74	12·4	,,

obtaining in actual coking practice. The effect of mass and comparatively prolonged coking period cannot easily be reproduced in the laboratory, although the method devised by Dr Lessing[1] gives useful and interesting data.

In his method the coal is heated electrically in a silica tube by means of a platinum resistance coil. A loose fitting silica tube weighted to any desired degree rests on the sample of coal. The swelling properties of a coal come out clearly by this method, and the nature of the residue is more decided than in the usual crucible test for the determination of volatile matter.

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[1] "A Laboratory Method for the Comparison of the Coking Properties of Coals." Dr R. Lessing, Inst. of Gas Engineers, 1912.

Attempts have been made by various observers to establish some connection between the action of various solvents on coals, and the coking properties of such coals. It is known that there is a diminution of the coking power of coals after extraction by pyridine, the effect being similar to that produced on exposing certain coals to the action of air for any considerable time. Extensive research on these lines has been carried out by Burgess and Wheeler.[1]

The results of certain of their experiments lead them to suggest that coal appears to contain two types of compounds, one type being more easily decomposed by heat yielding paraffins, the other type being more stable and yielding hydrogen. They consider the first type as most probably derived from the resins originally present in the sap of the coal plants (resin bodies), and the second type as a degradation product of cellulose (humus bodies). The "resin" bodies form the cement of a conglomerate, of which the cellulose derivatives are the base.

The weathering of coal depends upon the absorption of oxygen from air, and is detrimental to the coking of some coals, in some cases exposure for a week or two being sufficient to destroy their coking power. This property of oxygen absorption is most likely due to resin-like bodies, since it is well known that certain resins absorb oxygen. Bedson[2] proved that pyridine bases from coal tar dissolved 16 to 18 per cent. of a Durham coal, but had no action on anthracite, and Baker, working on similar lines, proved that coal after such extraction left a residue which no longer had coking properties. These results have been confirmed by other observers on various coals, and Bedson further proved that an amount of substance, practically equal to the volatile matter, can be extracted by pyridine. Burgess and Wheeler (*Chem. Soc. Trans.*, 1911) extracted from a Silkstone coal containing 33·4 per cent. of volatile matter, 30 per cent. of matter soluble in pyridine. It is probable that there is some relation between the coking properties of coals and their behaviour with various solvents. In some coals a resinous constituent appears to predominate, usually in those of a more feebly coking nature. In other coals some of the constituents are soluble, while others, though of resinous nature, are not, and in such cases the coking quality is not so much affected by the extraction.

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[1] "On the Volatile Constituents of Coal." Burgess and Wheeler, *Jour. of Chem. Soc.*, April 1911.

[2] *Journ. Soc. Chem. Ind.*, 1908, 147.

Much useful work on a laboratory scale has been done on the above lines, but although interesting results have been obtained, as yet they cannot be considered conclusive, and no hard and fast rule can be laid down as to the behaviour of a coal in a coke oven as determined by volatile matter, ultimate analysis, or results of extraction.

Most coke oven firms are prepared to subject samples of coal to coking tests on a "manufacturing" scale in which the effect of disintegration, compression, moisture, duration of coking period, etc., can be observed in an ordinary full-sized oven, and only such tests can be considered reliable. There is, however, a wide field for research on the chemistry of coal, and it is interesting to note that the British Association Committee on Fuel Economy, realising the importance of this branch of fuel technology, has appointed a "Chemical and Statistical" sub-committee to deal with the question. Some of the members have undertaken experimental work on the constituents of coal, their mutual relations, and their influence upon the character of the products obtained by distillations or oxidation.

The members of this sub-committee are of the opinion that the time is ripe for the organisation of a scheme of systematic co-operative research aided by national funds.[1]

The authors heartily endorse this opinion and express the hope that some tangible result of their deliberations may yet be seen in the form of a central national fuel laboratory in which the behaviour of coal in full-sized retorts and coke ovens, worked by practical engineers, may be observed in conjunction with research in a laboratory fitted with the necessary equipment for this class of work. In many cases the coal arriving at a coke works is fairly uniform in character and composition, but in some cases, especially when the coal comes from several collieries, a wide variation is met with. In some cases, a certain proportion of moderately coking coal may be mixed with superior coking coals. Thus a rich coal is sometimes mixed with a lean coal or vice versa, whilst a proportion of pitch under certain conditions may be utilised in this manner, sometimes to the advantage of the coke.

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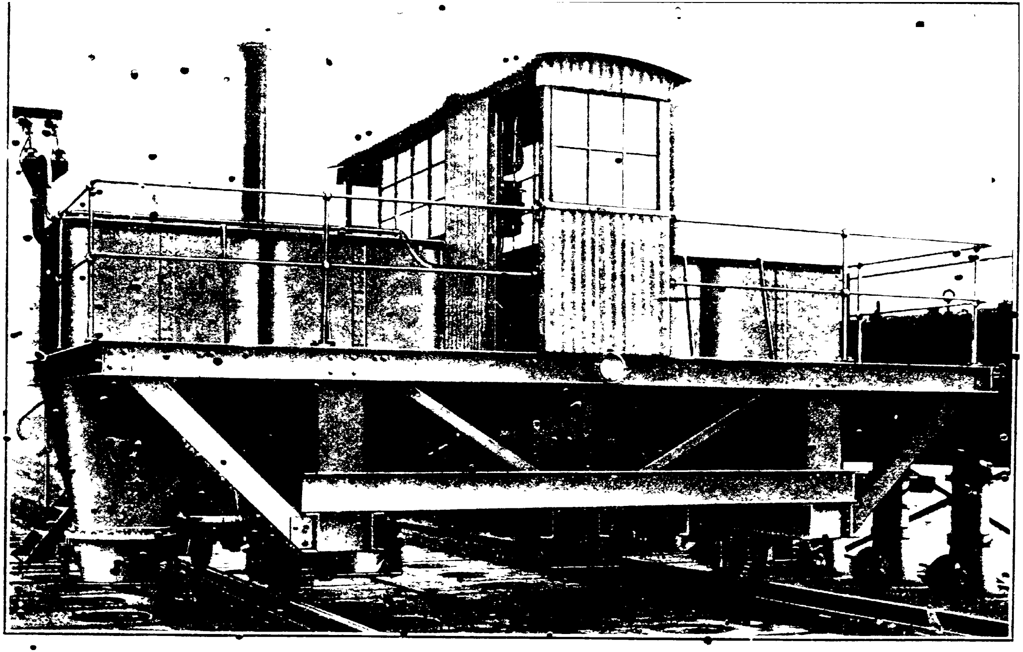
[1] Report of British Association Committee on Fuel Economy. Abstracted in *Jour. Soc. Chem. Ind.*, 16th October 1916.

It is highly important that in these cases the coal be thoroughly mixed. For instance, the coal from one district may be relatively high in volatile matter, tending to give a weak porous coke, whilst that from another district may be deficient in volatile matter. In mixing such coals a mere adjustment of the order of the wagons at the main tip is not sufficient. It has been the writer's experience that, in spite of various apparent opportunities of mixing in the disintegrators, bunkers, conveyors, etc., one wagon load of doubtful coal tipped between wagons of good material will not disseminate itself satisfactorily. The ill effect will be localised in one or two of the ovens, and the influence on the character of the coke will be obvious. A more satisfactory method would be to deliver the two qualities into separate low-level chambers, each having its own elevator into the disintegrator or bunker as the case may be. The proportions of the two qualities can then be controlled by regulating the relative speeds or capacities of the elevators. The next step is the charging of the oven. In some cases the ovens are charged in a similar manner to the beehive ovens, by means of tubs, feeding the oven through three or four charging holes in the roof. The use of individual tubs has now largely been replaced by the modern charging car, one type of which being shown in Fig. 47. Generally speaking they are electrically driven, hold a complete charge, and can be worked by one attendant.

In such cases the surface of the charge is uneven, consisting of a series of cones and depressions, restricting the passage of the gases evolved. Consequently the charge is levelled by hand or, as is more commonly the case, by means of a levelling bar attached to the ramming machine, as in Fig. 48.

Whilst these methods of charging are satisfactory in many cases, instances arise in which it is advisable to compress the charge after a previous disintegration of the coal. This combination is decidedly advantageous with coals of low



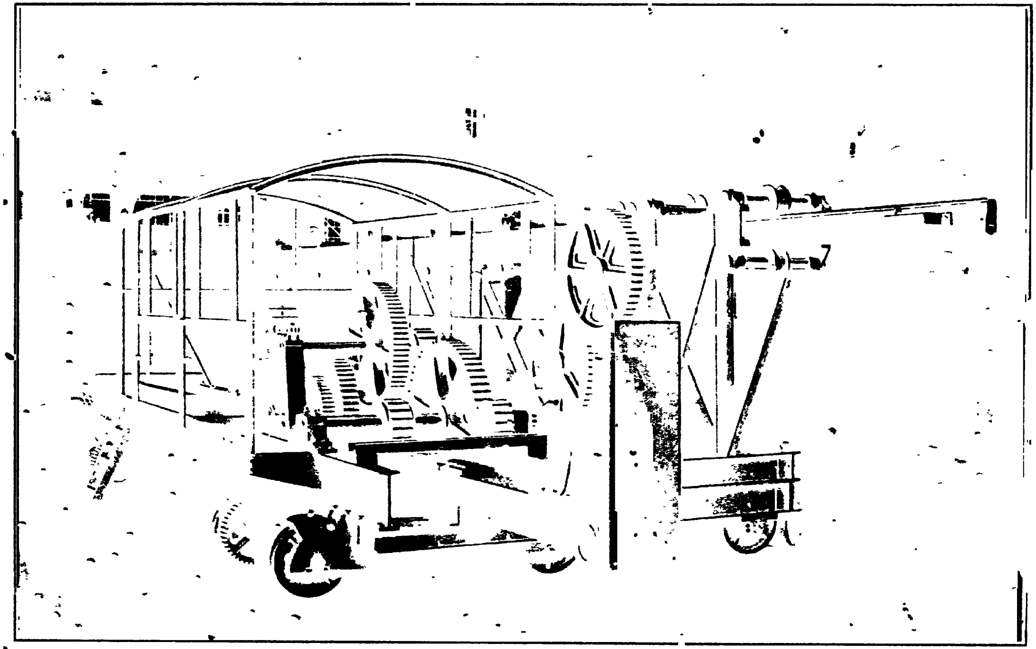


Fig. 48.—Buchanan's Ramming and Levelling Machine.

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volatile matter. The disintegration lessens the spaces between the particles whilst the compression forces the particles still closer together. Thus, by reducing the gaps, the comparatively weak binding tendency is utilised to fuller extent. Again coals with high volatile matter tend to give porous coke. Larger particles, and consequently larger air spaces between them, tend to accentuate the porosity. Disintegration and compression increase the density of the charge with a corresponding improvement in the coke. This is clearly shown in the example given below, the figures being from a case under the writer's observation:—

	I.	II.
Volatile matter in coal - -	28·5 per cent.	28·5 per cent.
Weight in lbs. per cubic foot:—		
Disintegrated, but not compressed - - - -	46·13	49·7
Disintegrated and compressed —		
(*a*) Top of charge - -	56·73	56·70
(*b*) Middle of charge -	56·84	58·11
(*c*) Bottom of charge -	58·93	60·80
Average increase in density -	21·6 per cent.	17·7 per cent.
Moisture in coal - - -	11·4 „	9·1 „
Coke made:—		
Average apparent specific gravity - - - -	1·110	
Average real specific gravity -	1·841	
Average pore space - -	39·7 per cent.	
Crushing strength - -	2,050 lbs. per square inch.	

The crucible test in the laboratory would lead one to expect a decidedly weak, swollen, and porous coke. The excellence of the coke produced as above in the coke oven shows the comparative unreliability of the laboratory test.

The advantages derived from the use of compressed charges may be summed up as follows:—

1. Output increased at least 10 per cent.
2. Denser coke.
3. Amount of coke breeze reduced.
4. Saving in labour.
5. Less wear on oven linings

The following is a general description of the type of compressing plant introduced into this country by Mr J. H. Darby:—

The slack from the washer is first crushed in a Carr

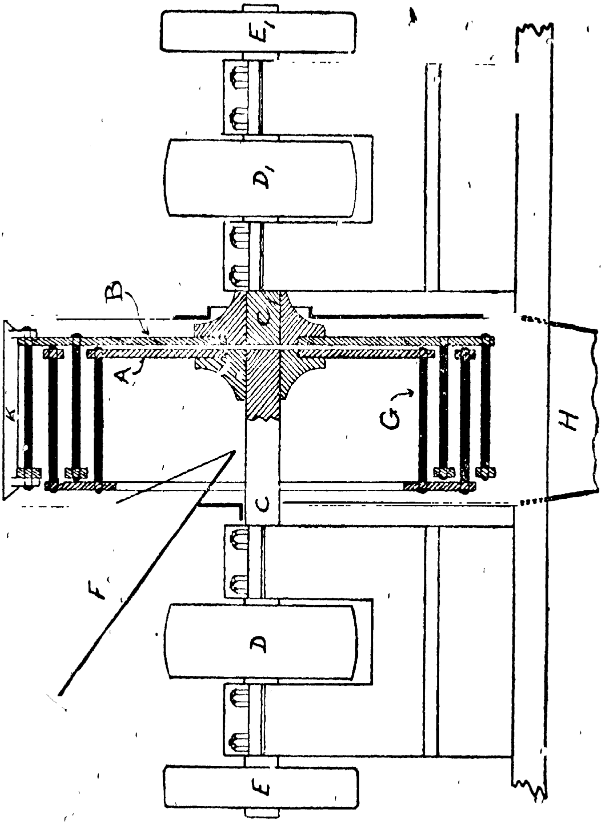


FIG. 49.—Carr Disintegrator.

disintegrator (Figs. 49 and 50). This consists of two reels A and B mounted on the shafts C and C_1. Each reel consists of two concentric rings in which steel spindles, of round or square section, are fixed from 2 to 3 in. apart. The reels

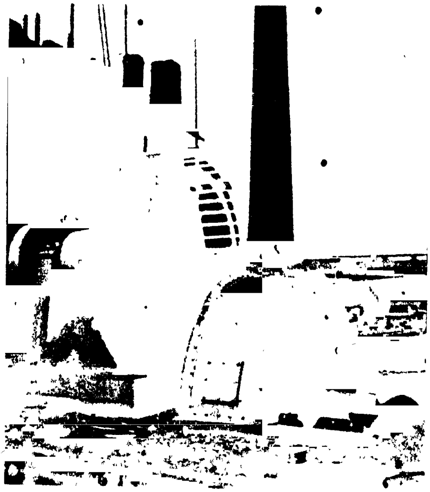


FIG. 50.—CARR DISINTEGRATOR WITH CASING REMOVED.

are driven by the pulleys D in opposite directions, and an even running is secured by the flywheels E giving the correct balance.

The slack is fed from elevators and the shoot F into the centre of the reels, and passes through the bars of the reels at G. As these bars are revolving in opposite directions at a peripheral speed of 6,000 ft. per minute, the slack is crushed to a fine state of division, and passes down the shoot H. The crushed slack is taken from this shoot to a storage bunker, usually situated near the ovens. From thence the crushed slack is delivered by means of a conveyor belt A (Fig. 51) into the box B of a charging machine. This box is shaped somewhat similarly to the interior of the coke oven, but the width of the charge can be adjusted by the patented arrangement for opening or closing the sides, consisting of eccentrics D, and shafting controlled by one hand-wheel. As the slack is fed into the box it is compressed by the weighted pole F of the stamper E, which traverses the whole length of the box. The pole is raised by an eccentric friction wheel G, driven by an electric motor, which also, by means of suitable gearing, causes the machine to move forward a predetermined distance during the up-stroke of the stamping pole. The length of fall of the stamper pole is constant, adjusting itself as the charging box is filled. Whilst a single stamper will compress a charge satisfactorily, it is an advantage to have two stamping machines, which allow of better compression, saving in time, and less dislocation to the plant in general should one of these stampers break down.

The drawing shows the charging machine separate and distinct from the ramming machine, but for smaller batteries of ovens it is more economical to use a combined ram and charger, as the lesser number of ovens to be charged per day allows ample time for the charge to be stamped, and then kept waiting until the contents of the oven are discharged by the section of the combined machine containing the discharging ram. When the compression of the charge is completed, the machine travels to the oven requiring a charge, the sides of the box are slightly opened by a hand-wheel, and the charge is carried into the oven by the peel A (Fig. 52) which forms the bottom of the box. This peel is fitted with a rack underneath into which fits a pinion driven by an electric motor and suitable reduction gearing. Most stamping machines work on the same principle. The pole is raised by various methods,

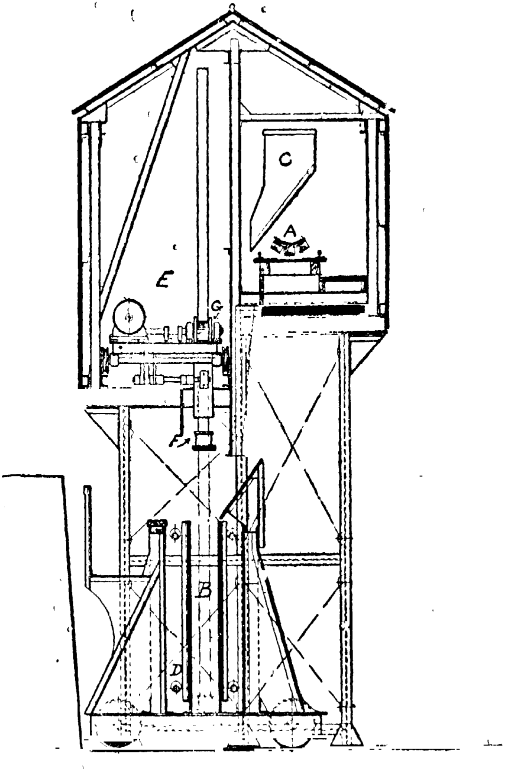


FIG. 51.—Arrangement of Coal Stamping Machinery.

frictional, compressed air, electro-magnetic, etc., and allowed to drop freely.

In the Meguin type of stamper (Fig. 53) the "pole" is replaced by a steel rod, which is raised by means of a crank and connecting rod. On the upward stroke the rod is gripped by the tong arrangement shown. Towards the end of this

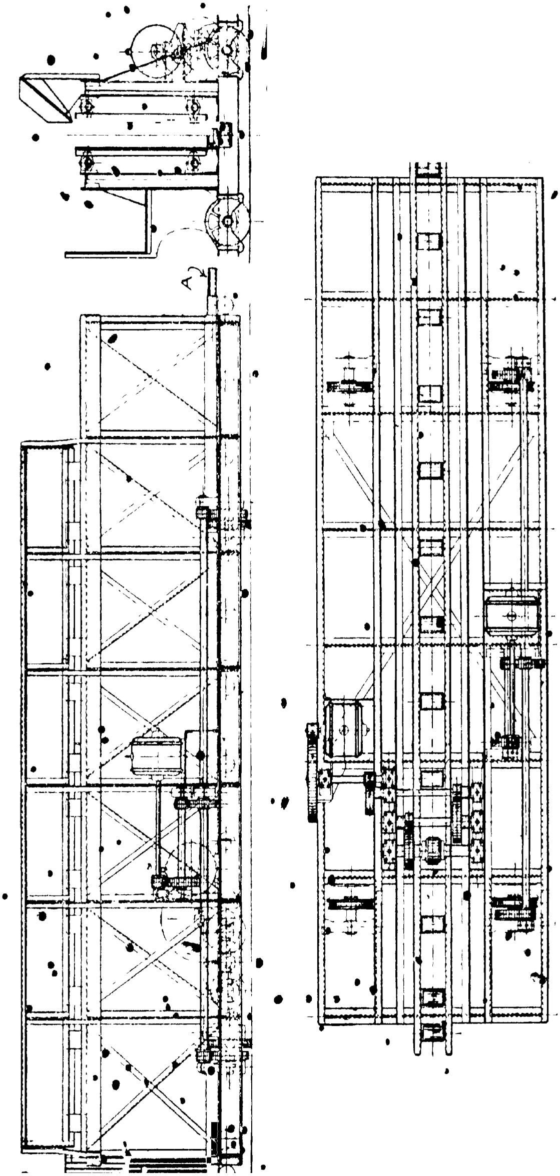


FIG. 32.—DARBY COMPRESSING MACHINE.

stroke the "tongs" come in contact with lugs, and are thereby opened, allowing the rod to drop freely.

Another modification, by the same firm, is shown in Fig. 54. In this the stamping piston is actuated by the conjoint effect of pressure and vacuum, the stamping cylinder A being coupled direct to the compressing cylinder B. The speed of this machine is much greater than the former, and the capacity is considerably increased. A later type has a separate air reservoir, rendering the motion of the compressor independent of the stamping motion. Fig. 55 shows the type of stamping machine manufactured by Messrs Bever, Dorling, & Co., Bradford. The stamping pole is elevated by means of two pairs of cams covered with "ferodo." The pressure of the cams can be adjusted by tension screws, whilst the cams are designed to allow the "ferodo" to be readily replaced when necessary.

The appearance of the coke is a very important matter. Formerly the coke was pushed out by a ramming machine on to a horizontal paved floor, where it was quenched by means of an ordinary hose pipe. The incandescent coke, being thus exposed to the atmosphere, suffered through oxidation before being thoroughly quenched, giving rise to a dark appearance in the coke. In extreme cases the ash left after combustion of the surface of the coke was obvious, giving the coke a reddish-brown tinge. Coke should be quenched quickly, avoiding contact with air as much as possible, and an apparatus on the lines of the Darby quenching hood, shown in Figs. 56 and 57, is advisable. In this coke-quencher the prism of coke is shielded from the air by the side plates E, whilst simultaneously a copious supply of water under considerable pressure is sprayed on to the coke as shown.

An efficient development of this idea is shown in Figs. 58 and 59 (Goodall's combined quenching, screening, and loading machine). "The machine consists of a revolving table, with vertical side plates, and two distinct operations are involved in the use of it. The first operation, quenching, is carried out by pushing the coke on to the slowly revolving table. Here it is spread out by the action of the table, and quenched by water sprays.

The tall sides of the chamber prevent undue access of air.

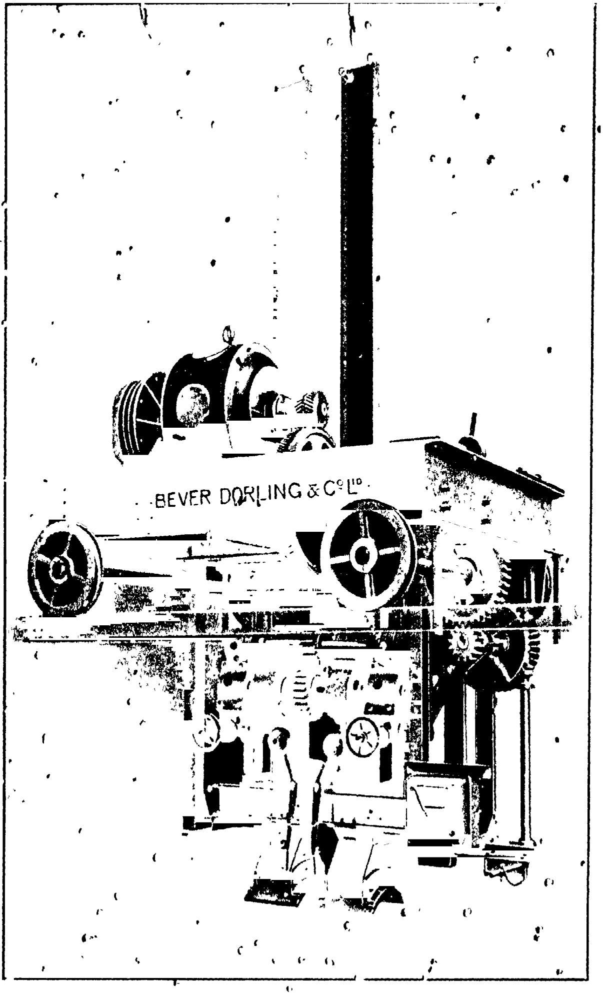


FIG. 55.—STAMPING MACHINE. (Bever, Dorling, & Co., Bradford.)

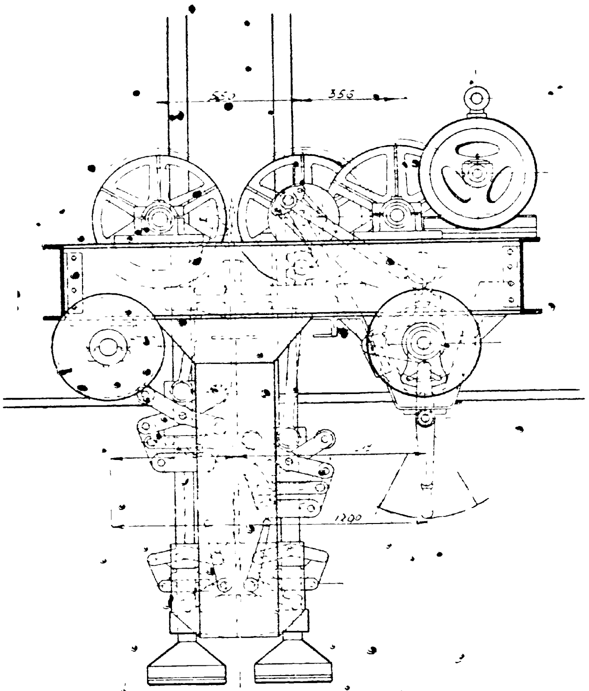


FIG. 53.—MEGUIN STAMPER.

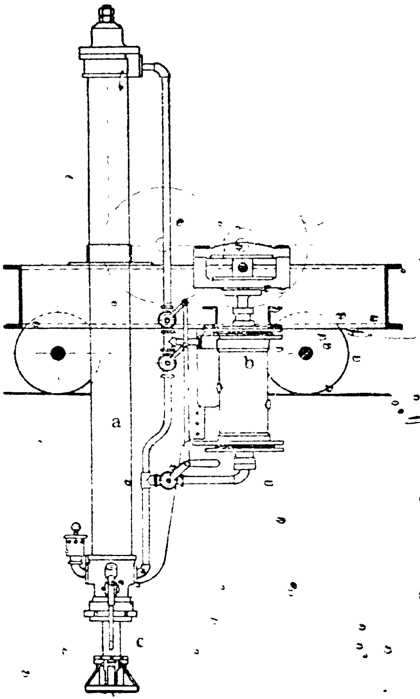


FIG. 54.—MEGUIN STAMPER. (Compressed air ty

and an atmosphere of steam is maintained until the coke is completely quenched. In the second operation the table is revolved in the opposite direction, and the contents are scooped off the table by a deflecting plate over a reciprocating screen, and delivered direct into the wagon as screened coke.

Other methods of dealing with the coke are shown in Fig. 60. Methods of dealing with the coke rapidly by mechanical methods have multiplied considerably of late years, and the instances shown are merely typical. The type chosen for any particular plant will depend on the local circumstances. Thus, in some cases where the plant is not favourably situated for carrying out repairs to mechanical devices, a simpler type, such as Fig. 60*a* or Fig. 60*b*, is preferable. In cases where the coke is sold in the open market mechanical screening as shown in Fig. 44, combined with efficient quenching, is highly desirable, whilst in some few cases the coke is required for blast furnaces in the immediate vicinity.

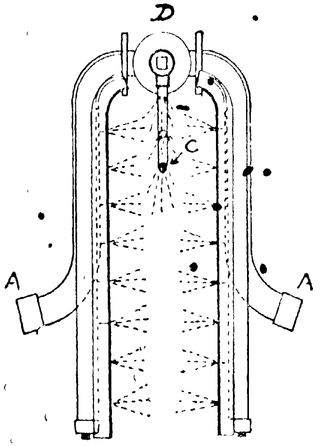


FIG. 56.—Darby Quenching Hood.

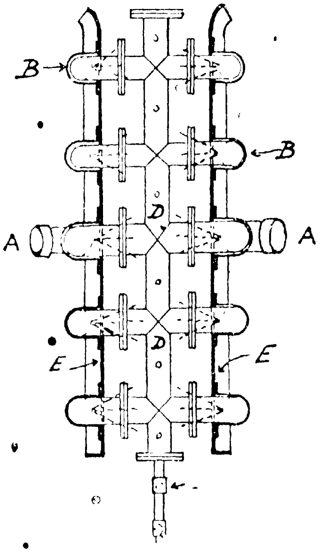
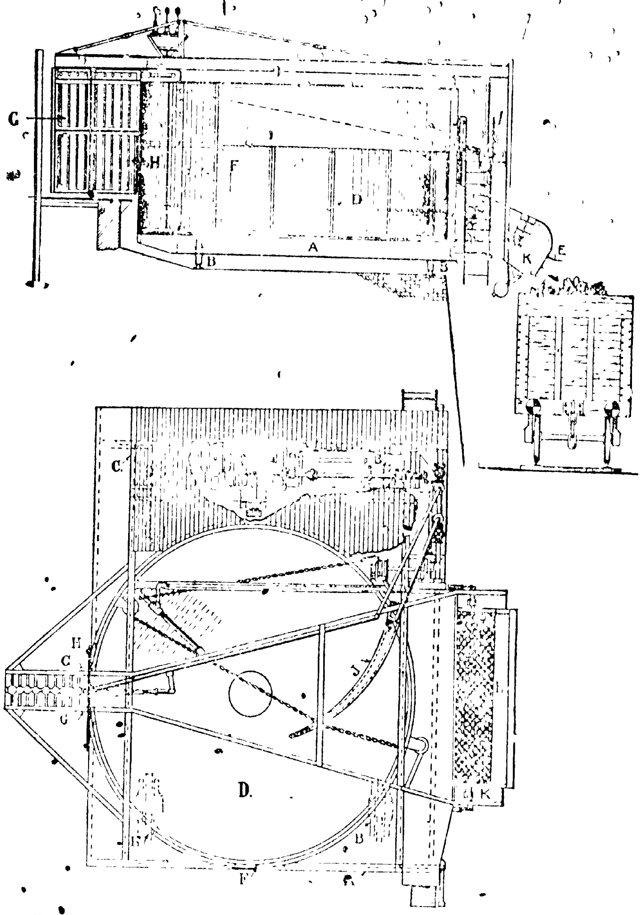


FIG. 57.—Darby Quenching Hood.



Figs. 58 and 59.—Goodall Quenching, Screening, and Loading Machine.

Under these conditions a sloping bench delivering into the furnace barrows or into ropeway skips (Fig. 61) is a convenient type to adopt.

Fig. 62 shows a complete arrangement of mechanical appliances in connection with a Koppers coke plant.

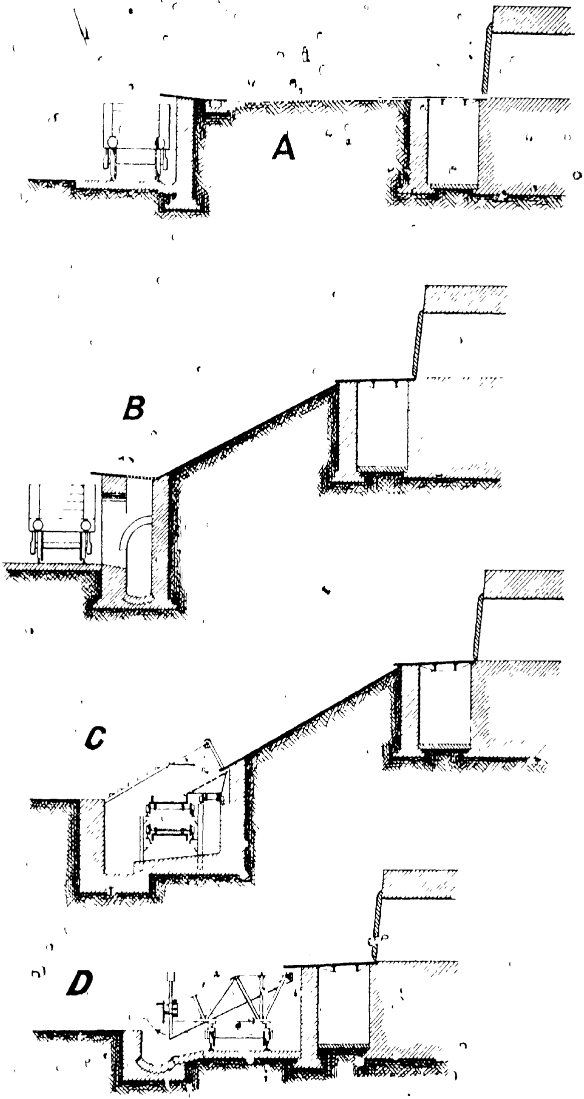


Fig. 60.—Methods of Handling Coke.

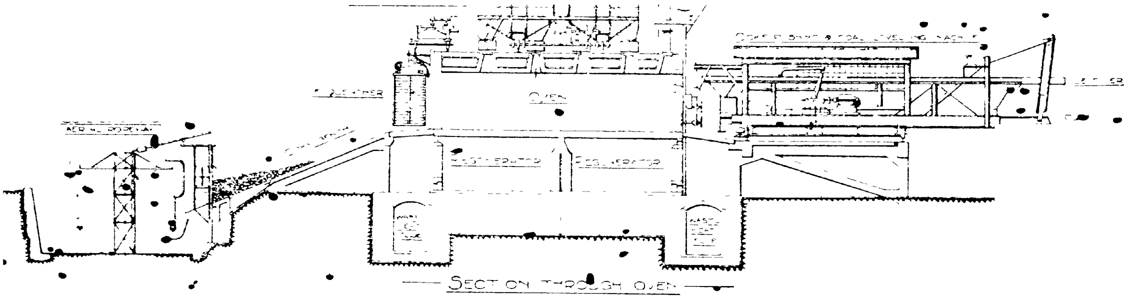


Fig. 61.—Mechanical Devices at a Koppers Coke Plant.

CHAPTER XI

CHEMICAL AND PHYSICAL EXAMINATION OF COKE

IN this country a complete chemical and physical examination is rarely carried out. The former is usually confined to an estimation of ash, sulphur, moisture, and sometimes volatile matter, and the latter to a visual examination of the appearance of the mass of coke or of individual pieces. Occasionally the nitrogen content is sought, not as a matter of concern between buyer and seller, but as being a useful point in considering the yield of ammonia. In some rare cases the iron content is estimated, its effect on the clinkering properties having been already commented upon. Of recent years the phosphorus content has received more serious attention. In the blast furnace the phosphorus tends to pass into the iron, lowering the elasticity and rendering the metal more brittle. In general the phosphorus content of the coke is low enough to remove all misgivings, but cases may arise in which the effect of phosphorus is not altogether negligible. In a few isolated instances the presence of arsenic is injurious as in the case of malt kilns.

The estimation of ash, sulphur, moisture, volatile matter, and nitrogen have already been discussed in Chapter V., but we might at this stage emphasise the importance of ensuring that the coke be thoroughly burnt off in the case of ash and sulphur and that the coke be finely powdered in the case of nitrogen estimation, and allowed sufficient time for complete decomposition.

Iron is estimated by burning off a sufficient quantity of coke to give a workable amount of ash. Obviously, assuming the coke to contain 10 per cent. of ash, it will be necessary to incinerate 10 grm. of coke to obtain 1 grm of ash. The ash, after the prolonged ignition necessary to obtain a sufficient quantity to work on, is not readily soluble, as regards its iron content, in dilute hydrochloric acid. In some instances even concentrated acid requires a considerable time to completely dissolve the iron oxide. When such is the case fusion must be resorted to, or treatment with hydrofluoric acid, as described under phosphorus estimation. Finally, when complete solution of the iron is effected, the ferric chloride is reduced by means of stannous chloride and then titrated with a standard solution of potassium bichromate in the usual manner.

Estimation of Phosphorus.—One gram of the ash is weighed, transferred to a beaker, and boiled gently with concentrated hydrochloric acid for about two hours, at the end of which time complete solution of ferric oxide and phosphates will have been effected. If, however, the insoluble residue is still reddish or brown in colour, more hydrochloric acid must be added, and the digestion continued.

It is fairly certain that unless all the ferric oxide is dissolved, that some of the phosphorus will remain with it as phosphate. If, as may sometimes be the case, the ash resists decomposition after prolonged digestion in the acid, then other means of attacking it must be used, such as fusion with alkaline carbonates, or treatment with hydrofluoric acid.

In the former case, 1 gm. of ash, previously ground fine in an agate mortar, is mixed intimately with 5 or 6 gm of fusion mixture (potassium carbonate, 138 parts; sodium carbonate, 106 parts) and transferred to a platinum crucible, covered with a platinum lid, and placed in front of a gas muffle for ten to fifteen minutes, then gradually placed further in the furnace until it attains the full heat. Gradual heating is necessary to prevent frothing up and loss of the contents. The mass fuses and the fusion should be maintained for an hour. At the end of that time the crucible and contents are taken out and allowed to cool. They are next extracted with hot distilled water and transferred to a porcelain evaporating basin, taking care to avoid any loss of material. The basin is covered with a large clockglass, and hydrochloric acid cautiously added avoiding loss by effervescence of the carbonates. When the addition of acid no longer causes effervescence, rinse the clockglass into the basin, add a slight excess of acid, and evaporate carefully over a low temperature burner. Unless this is done cautiously, much loss, due to spitting of the salts, will occur. When evaporated to complete dryness, continue the heating for about half an hour to expel acid and to dehydrate the silica, the latter being the important point. Allow to cool, add a little strong hydrochloric acid, and warm gently for a few minutes, then add hot distilled water, digest five or ten minutes, and filter, washing the paper several times with hot dilute acid, finally twice with hot water. The filtrate now contains all the phosphates in solution. Add 2 or 3 c.c. of a solution of ferric chloride, then ammonium hydrate solution until the solution smells slightly of ammonia. Boil up and filter. The reddish precipitate consists of ferric hydrate and phosphate together with alumina. The object of this separation is to get rid of the alkaline chlorides resulting from the fusion mixture originally used. Wash the precipitate three times with hot distilled water. Remove the filtrate and replace an empty beaker under the funnel stem.

Carefully pierce the filter paper at the bottom of the cone with a platinum wire, or fine glass rod, and wash the precipitate through. Then dissolve by warming in as little hydrochloric acid as possible. The solution should be quite clear. Neutralise carefully with ammonium hydrate until just turbid, then add 10 c.c. excess. Now bring back with nitric acid until almost clear. The solution is then about neutral. Add 5 c.c. concentrated nitric acid, and bring the whole to the boiling point; then add 18 c.c. of a 10 per cent. solution of ammonium molybdate, and put aside in a warm place to settle, after a few minutes' shaking round. The volume of the solution finally ought to be from 50 to 70 c.c. If properly precipitated the yellow phospho-molybdate comes down in a granular condition, soon settles out, and is easily filtered off and washed, using cold water acidulated with nitric acid; six or seven good washings are necessary. Finally, take the filter paper and its contents from the funnel, open out flat and place on a flat tile or glass plate in the water bath, drying until constant weight is obtained.

The precipitate contains 1·63 per cent. of phosphorus, or 3·73 per cent. phosphoric acid. The fusion method as above described is somewhat tedious, but is occasionally necessary.

If a complete analysis of the ash is required, the fusion method of decomposition is the best one.

The use of hydrofluoric acid often saves time, especially when it is desirable to know the iron and phosphorus content only. One gram of the ash ground fine in an agate mortar is transferred to a platinum dish, moistened with dilute sulphuric acid, and about 10 c.c. of hydrofluoric acid added, and digested at about 100° C. for an hour. In most cases complete decomposition of the silicates will have taken place in this time. Evaporate carefully to dryness on a hot plate. The residue will then dissolve in concentrated hydrochloric acid, and the solution thus obtained may be used for the determination of either iron or phosphorus. It should be noted that some hydrofluoric acid contains iron, and not improbably some phosphorus as well, so that a blank determination of these elements should be made, which, of course, only requires to be done once, so long as the same lot of acid is in use.

Arsenic in Coal and Coke.—The discovery of arsenic in beer brewed from malt and hops led to the source of the arsenic being traced to the fuel used in drying the malt. The arsenic is usually associated with the pyrites present in the coal, and the fact that coals may contain arsenic was pointed out by Percy in 1876. Dr Angus Smith proved the presence of this element in fifteen specimens of Lancashire coal, and it has also been found in German and French fuels, also in Nottingham and Newcastle coals. The accurate determination of minute quantities of arsenic in a substance such as coke presents very considerable difficulties. Descriptions of methods advocated, and discussions on same, are to be found in the *Journal* of the Society of Chemical Industry, May 31, 1901, p. 437 (Smith and Jenks), May 31, 1901, p. 449 (Archbutt and Jackson); and in the *Analyst*, 1901, **26**, 253-256 (Chapman).

Physical Properties of Coke.—Though in general an experienced eye can give a reliable judgment on the physical qualities of a coke, sometimes for comparative purposes a series of physical and mechanical tests offers useful data. A complete examination on these lines would comprise a test for apparent and real specific gravity, and from the respective results a calculation of the relative proportions of coke substance and pore space. Mechanical tests on the hardness and resistance to pressure would be included along with some form of "shatter" test to give a comparative index of the friability.

Apparent specific gravity, or the specific gravity of the coke as made, that is, coke substance plus air spaces, may be estimated as indicated in Chapter V., but a type of apparatus more suited for coke is shown in Fig. 62, which is in principle a volume meter.

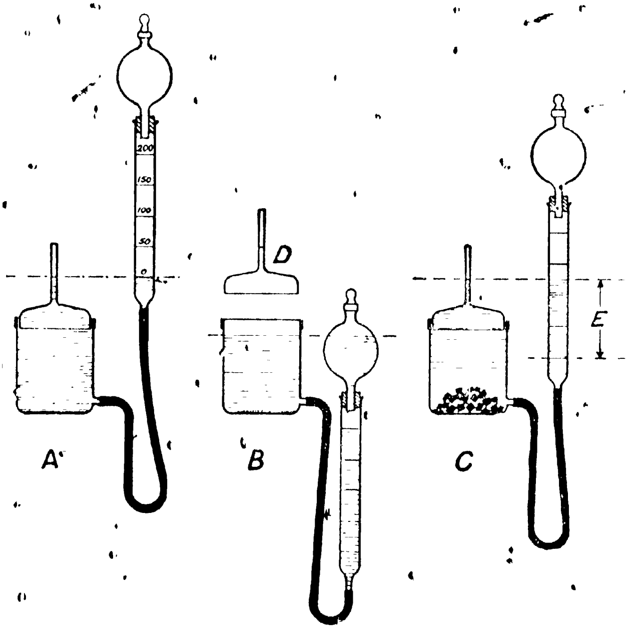


Fig. 62.—Apparent Specific Gravity of Coke.

A wide-mouthed vessel is fitted with a ground-in stopper having an extension in the form of a tube D. This vessel communicates by means of the flexible tube with a measuring burette, the upper end of which is fitted with a receiver. The relative positions of these vessels are shown in three stages. In the first stage A the stopper is inserted and water is run into the vessel through the burette until the water is at a specially marked level filed on the tube of the stopper, and at zero in C. In the second stage B the burette is lowered, thus bringing down the level of the water to allow of the removal of the stopper without loss of water. In the third stage the weighed sample of coke, in the form of well-dried pieces, fairly uniform in size, and brushed free from adhering coke dust, is admitted into the vessel. The stopper is tightly reinserted and the burette raised until the water is forced to the level of the mark. The vessel is shaken briskly to detach adherent air bubbles, and the reading on the scale of the burette is taken. The number of cubic centimetres of water E gives the weight of water displaced by the bulk of coke in the sample. The test should be carried out as expeditiously as possible to avoid undue access of water into the pores of the coke.

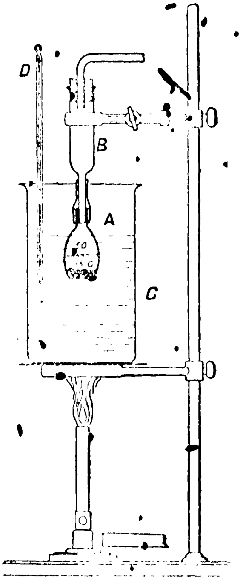


FIG. 63.—Real Specific Gravity of Coke.

Real specific gravity, or specific gravity of the coke substance, is a little more difficult to estimate. In general, this is carried out by means of some form of specific gravity bottle. The coke must be finely ground to break up all the cells and liberate the air and gases, the removal of which is facilitated by a partial vacuum.

A suitable apparatus is shown in Fig. 63: A is an ordinary 50 c.c. specific gravity bottle. B is an adapter which allows easy connection with a vacuum or filter pump (Bunsen water pump), serving also as a catch box for any particles of coke carried up by excessive ebullition. This adapter temporarily replaces the perforated stopper, and should be connected with rubber tubing in the manner shown, or preferably ground in. C is a water bath. Three grams of finely powdered coke is placed in A, and covered with about an inch of water. The apparatus is fitted up, and the water bath kept at about 90° to 100° C. The vacuum pump is started and maintains a suction in the vessel A, the contents of which are kept gently boiling for two or three hours. The thermometer, D will give an indication of the vacuum in A owing to the reduced boiling point. The adapter is washed out, if necessary, into the specific gravity bottle and removed. The bottle is allowed to cool and is then filled with distilled water, utilising its own perforated stopper, and weighed. The calculation of the result is as follows:—

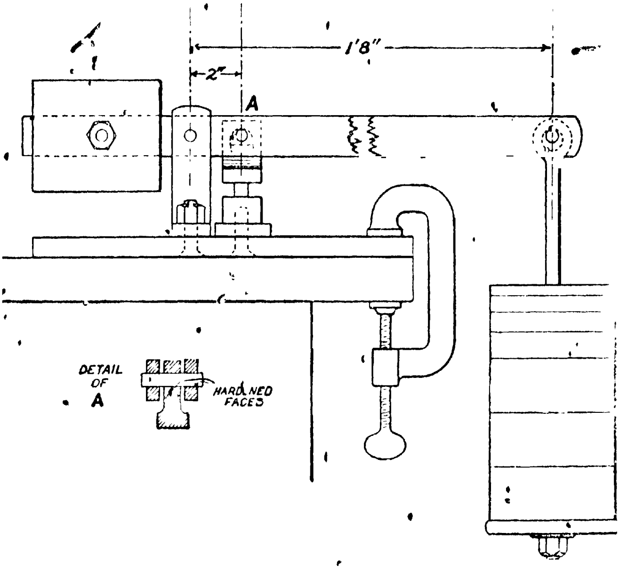


Fig. 64.—Compression Testing Machine.

Weight of bottle, plus sample, plus water content of bottle - - - - - - - -
Weight of bottle, plus sample, plus water to fill, *i.e.*, final weighing -

Difference (x) = weight of water displaced by 3 gm. of coke substance.

Then $3 \div x$ = real specific gravity.

The proportion of coke substance is then found, thus:—

1. $\dfrac{\text{Apparent specific gravity}}{\text{Real specific gravity}} \times 100$ = per cent. coke substance.

2. $100 -$ per cent. coke substance = per cent. pore space.

The hardness, or more correctly the compressive strength, is found by means of an ordinary compression testing machine. A simple type that can be made by the average coke works mechanic is shown in Fig. 64, and has been used by the author to good effect. It is not claimed that this type is strictly accurate, but its results are certainly comparative. When we consider that the longest piece of coke from a retort oven is only half the width of the oven, and that the crushing strength will vary even in this short length, we see that even with an elaborate machine considerable variation in results can be introduced by incorrect sampling. In the machine shown, clean pieces of coke are ground to approximately half-inch cubes, placed between the faces of the crushing section, and weights gradually added at the end of the lever until collapse of the specimen takes place. The pieces must be free from inherent flaws or cracks, and several pieces from a batch of coke must be tested and an average taken. The weight of the lever is balanced, and the position of the pin is designed to give a leverage of 10 : 1,

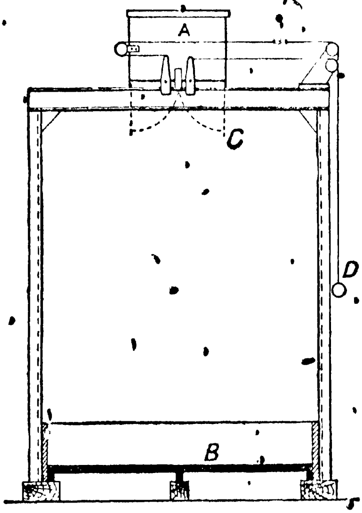


Fig. 65.—Shatter Test for Coke.

so that the weights added, × by 10, and corrected to a sectional area of 1 sq. in., give the desired result. In some cases, whilst small individual pieces may show a satisfactory compressive strength, the batch as a whole is inclined to break into small pieces or fingers. The effect of this in a blast furnace would be quite as serious as a low compressive strength, and various devices have been designed to give correct comparative tests on the friability of coke. A good method, shown in Fig. 65, has been adopted by the Bureau of Mines, U.S.A. This consists of a box A holding 100 lbs. of coke mounted on a support, the bottom of the box being 6 ft. above the cast-iron shatter plate B. The bottom doors C are released simultaneously by the cord D, giving a free fall for the coke.

A weighed sample is placed in the box, dropped four times on the cast-iron plate, the small material and dust being returned to the box each time. After this the coke is screened on a 2-in. mesh screen, the proportions passing through giving a relative comparison of the friability.

C. A. Meissner, U.S. Steel Corporation,[1] states that good physical tests for a standard quality of blast furnace coke are shown by the following:—

	Shatter Test.		Specific Gravity.		Porosity per Cent. Cell Space.
	Through 2-in. Screen	Over 2-in. Screen	Apparent.	Real	
1	15·16	84·84	·976	1·841	47·03
2	16·11	83·89	·950	1·824	47·92
3	14·06	85·94	·992	1·831	46·31

The writer would suggest the following as a good specification for coke:—

Ash,	not over	10	per cent.
Moisture,	,,	3	,,
Sulphur,	,,	1	,,
Phosphorus,	,,	·02	,,

Pore space, 40 to 50 per cent. (furnace coke).
Pore space, 25 to 40 ,, (foundry coke).
Compressive strength, over 1,200 lbs. per square inch.

---

[1] "Modern By-product Coke Ovens." C. A. Meissner, American Iron and Steel Institute.

APPENDIX

VOL. I.

THE Simplex ovens are designed with vertical flues as a rule, but where it is desired to instal ovens of the utmost simplicity and ease of working, a horizontal flued type is recommended, the general arrangement being shown in Fig. 66. The heating gas returns by the two delivery pipes.

All the air which is necessary to burn the total quantity of gas enters in the flues A together with the gas supplied by the burners No. 1, which burners are regulated according to the temperature it is desired to keep in the flues A.

A second supply of combustible gas is given by burners No. 2, the regulation of which affects the temperature in the flues B.

A third supply of combustible gas is given by burners No. 3, the regulation of which affects the temperature in the flues C.

Lastly, a fourth supply of combustible gas is delivered from the burners No. 4, which regulates the temperature in the flues D.

It will be seen that each burner controls the temperature of a different flue, perfect control of temperature is obtained, and the said temperature can easily be kept as low as is consistent with good coking in the upper flues A, and made to increase gradually in the flues B, C, and D, so as to obtain a maximum yield of by-products.

Each oven wall is provided with a single air inlet on the coke bench side, to the exclusion of any other air inlets. This causes the working of these ovens to be unaffected by the direction and strength of the wind.

The construction of the walls is such that their strength is equal to that of vertical flued walls of similar height and width, whilst on the other hand the facilities for inspection, easy regulation, and the thickness of the walls make this type of oven an ideal one to be worked without skilled labour and with a minimum of supervision.

All blocks are tongued and grooved, of sufficient thickness to prevent contraction when charging the ovens, thus avoiding any leakage of gas between the coking chambers and the flues during the whole time of carbonisation.

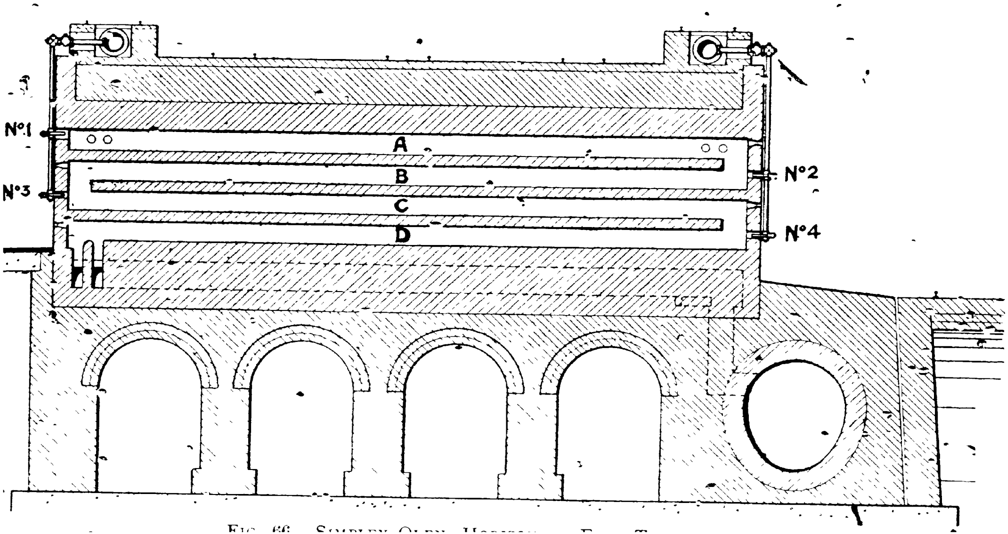


Fig. 66. Simplex Oven. [illegible]

In the Simplex vertical flued waste heat type of oven (Fig. 67), every other vertical heating flue is uniformly heated by ascending burning gases, whilst the products of combustion descend in the adjacent flue. This construction ensures correct proportions of air and gas, and in conjunction the number of gas burners (14, 15, or 16 per oven) ensures a good distribution of the heat from one end of the oven wall to the other. The whole of the air for combustion, circulating through the oven foundations on its way to the heating flues, ensures a transference of most of the heat ordinarily lost by conduction, resulting in an appreciable saving of heating gas.

The method of heating the Simplex vertical flued waste heat ovens may be described as follows:—

The cold air admitted at the end of the gallery A is distributed by the gallery B to the vertical heating flues C and C_1, where it meets a regulated supply of gas distributed through the pipes D and E and the taps F and G to the burners H and H_1.

Under the influence of the chimney draught the burning flames ascend in the heating flues C and C_1 to the top horizontal flue I, and the products of combustion descend in the heating flues J and J_1, passing through the openings K and K_1 into the sole flues L, from which they reach the main flue N by means of the conduits M controlled by the dampers O.

In order to prevent formation of black ends an excess of air is supplied to the vertical heating flues C_1, which excess of air meeting an additional quantity of gas supplied by the pipes P, the taps Q, and the burners R ensures a secondary combustion taking place in the end flues J_1, so as to compensate for the loss of heat by radiation and thereby ensure that the ends of the coal charges are carbonised at a sufficiently high temperature.

Special attention is drawn to the fact that a uniform temperature is obtained throughout the oven walls with only one regulating gas cock per oven, which gas cock is quite accessible in the air distributing gallery A.

Inspection holes S are provided at both ends of the horizontal top flue I for the purpose of ascertaining that the temperature is absolutely uniform from end to end of the ovens.

The Simplex regenerative oven (Fig. 68) possesses many novel and desirable features. In the heating flues a system of fractional combustion is utilised to secure efficiency in this section, whilst the gas supply is constant, avoiding any necessity for reversing gas cocks, levers, and their attendant mechanism.

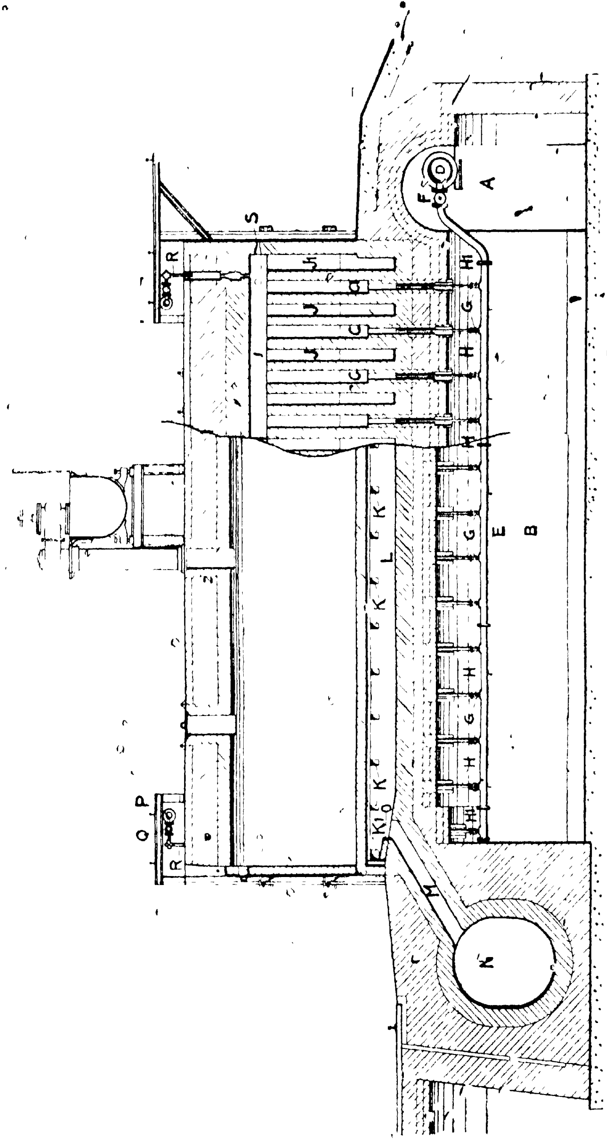


Fig. 67.—Simplex Oven. Vertical Flue. Waste Heat Type

The alternation of the direction of the heating gases is confined to adjacent flues, this subdivision naturally tending towards uniform heating of the oven walls. The advantage of accessibility to the gas cocks situated on the oven top will be obvious, and the position of the burners precludes all possibility of disturbance through falling particles of brick. The heating of this type is as follows :—

The cold air admitted by the general inlet main flue M is distributed by the conduits N into the regenerator A, from whence the heated air passes through the conduit B into the sole flue D. From this it ascends in the vertical heating flues F_1 and reaches the horizontal flue G, wherein it meets a first and regulated supply of gas from the burners H fed by the pipes I, J, and L. The air being in excess of the amount required to consume this gas, the burning gases and surplus air travel down the heating flues F_2, in which the first stage of the fractional combustion takes place, and are subsequently collected in the sole flues E, whence they ascend through the vertical heating flues F_3. The mixture of products of combustion and surplus air now reaches the flue G_1, where a second supply of gas is admitted from burners H_1. The gases and air now in correct proportion travel down the flues F_1, in which the second and final stage of the fractional combustion takes place, and are subsequently collected in the sole flue D_1 The final products of combustion pass through the regenerator A_1, the conduit [illegible], and the main flue P to the chimney.

A tap K is provided on each pipe J, in order to regulate the total quantity of gas supplied to the burners H and H_1, and so ensure correct proportions of gas and air for efficient combustion. When the regenerators A and A_1 have respectively given up or absorbed the requisite amount of heat, the draught is reversed. The first and second stages of combustion then take place respectively in the flues F_3 and F_1. The flow of burning gases is naturally reversed in all the flues, but the burners H and H_1, without any reversal, supply the requisite quantity of gas in a continuous fashion.

THE C.G.O. COMPOUND REGENERATIVE COKE OVEN

This oven has been specially designed with a view to effecting economies in coal and rich gas, and may be worked as an ordinary regenerative coke oven heated by a portion of its own gas, or as a "gas" oven. In the latter case the whole of the coke oven gas is available for external purposes, the oven flues being then heated by producer gas blast furnace gas, or a mixed gas. This type of oven is well

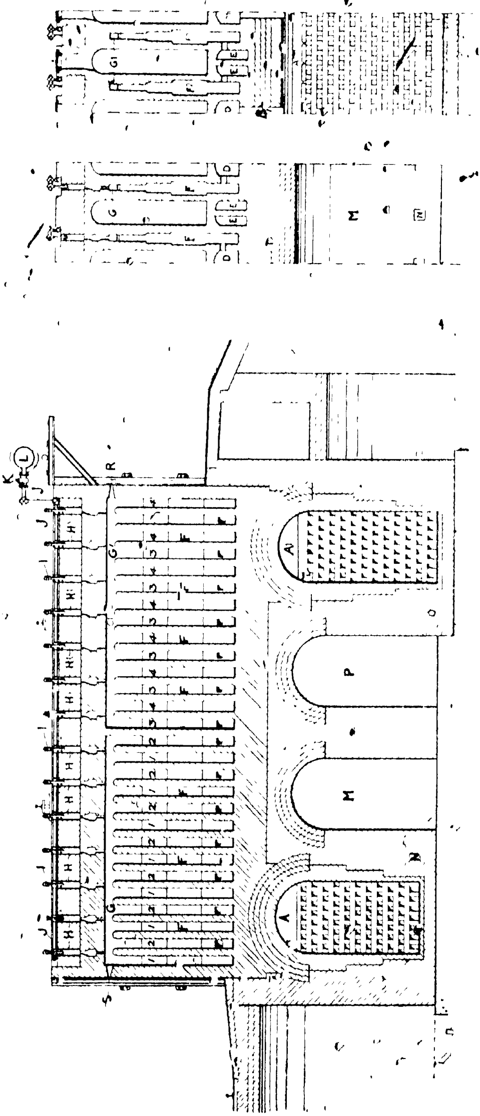


FIG. 68.—SIMPLEX REGENERATIVE OVEN

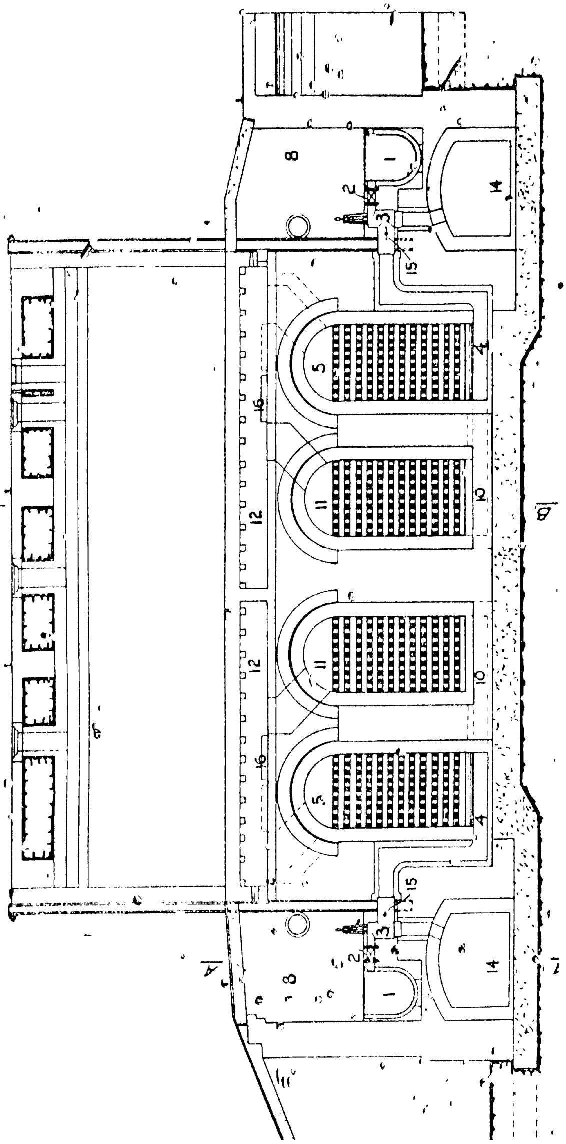
worthy of consideration in the case of iron and steel works, where supplies of blast furnace or producer gas may be used in the coke oven to good advantage, leaving the rich coke oven gas available for power, lighting, furnace heating, etc. In this type of oven any desired proportion of the ovens in a battery may be heated by coke oven gas and the remainder by poor gas. It will be observed that the regenerators of this oven are subdivided into two pairs, one pair being heated by the waste products of combustion from the flues of a dividing wall, whilst the individual components of the other pair are being used simultaneously to heat up poor gas and air respectively. The operation of the oven is as follows (Figs. 69, 70, and 71):—

A.—When heated with producer or blast furnace gas, the heating gas is taken alternatively from the gas mains 1 on the ram side or on the bench side of the ovens, and travels through the regulating cocks 2, the reversing valves 3, and the channels 4 to the regenerators 5, where the gas is heated by passing through the hot chequered brickwork. The hot gas enters into the flues 6, whence it is distributed into the combustion flues 7 The air for combustion is taken from the control passages 8 alternatively, and is drawn through the valves 9 and channels 10 to the regenerators 11, and after being heated enters into flues 12, whence it is distributed into the combustion flues 7 together with the hot gas. The air and gas mix and ignite at the bottom of the flues 7, and the products of combustion rise from the one-half of the oven walls and pass along the horizontal flues 13 into the vertical flues 7 of the other half of the oven walls, eventually splitting up into two circuits and being drawn by way of the flues 6 and 12 into the regenerators 5 and 11. After heating the chequer brickwork in the regenerators, the cooled gases pass by way of the channels 4 and 10 and valves 3 and 9 into the waste heat flue 14 which is connected with the chimney.

The travel of the air and gas is reversed every half hour. It will be noted that reversing valves 3 are connected to the poor gas main, whilst the valves 9 communicate with the atmosphere instead.

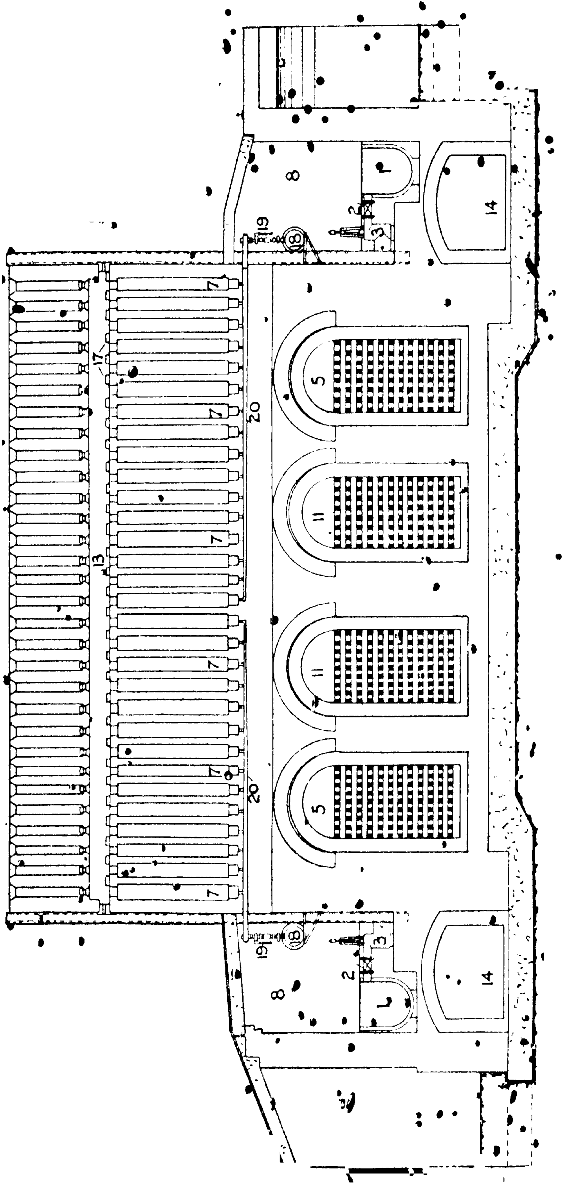
Regulation can be made by means of the butterfly valves 15, also by the damper bricks 16 and 17.

B.—When heated with coke oven gas the rich gas is taken alternatively from the gas mains 18 on the ram side or on the bench side of the ovens, and travels through the reversing cocks 19 to the channels 20, whence it is distributed to the base of the vertical flues 7. The poor gas is shut off from the mains 1, and air is admitted. This



Section through Carbonizing Chamber.

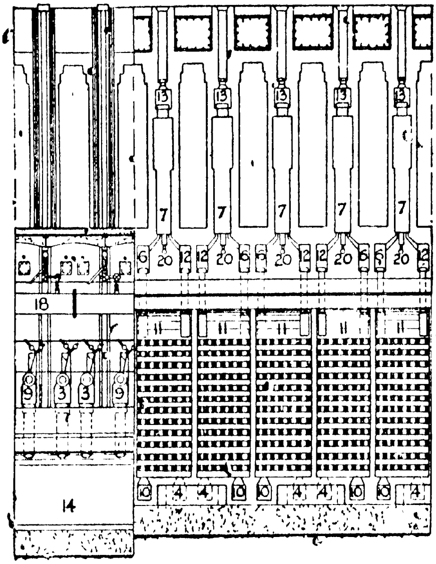
FIG. 69.—C.G.O COKE OVEN, COMPOUND REGENERATIVE TYPE.



Section through Heating Flues

FIG. 50.—C G O OVEN. COMPOUND REGENERATIVE TYPE.

air is drawn through the valves 3 and channels 4 to the regenerators 5. At the same time air is taken from the control passage 8 through the valves 9 and channels 10 to the regenerators 11. After being heated in the regenerators the air travels by way of the flues 6 and 12 to the base of the flues 7, where it mixes with the gas and ignites. The products of combustion travel over to the other half of the oven wall in a similar manner to that already described.



Section A-A. Section B-B.

Fig. 71.—C.G.O. Oven, Compound Regenerative Type.

The Coppée Co. have modified their type of oven in many respects by utilising the principle of individual regenerators, each oven with its own regenerators forming a self-contained unit. In addition, the flue system in the dividing wall is divided into two symmetrical sections, each working separately. These two respective sections work in conjunction with their own particular regenerators and sole flues, so that the reversing of the gas and air currents affects only one-quarter of the length of the side wall. Each regenerator is divided by a vertical partition, extending the full height of the regenerator, into sections which are respectively connected with their corresponding section of the heating flues. The system is shown in diagram form in Fig 72, in which the two left-hand regenerators (quite independent and distinct) are linked up to sections 1 and 3 of the heating flues, whilst the right-hand sections connect with the flue sections 4 and 2. Gas is distributed to the flues by means of two pairs of removable distributing "cornues" or channels, perforated with apertures carefully proportioned to allow equal distribution of gas in each flue. Each individual cornue feeds one-quarter of the oven wall, whilst each individual flue is provided with a damper at the top.

The special arrangement of the cornues and dampers and the very great accessibility, together with the system of regulating cocks, provide for a perfectly equal distribution of gas over every part of the side wall. Fig 73 shows the general layout of the Coppée individual regenerative oven. The air necessary for combustion is drawn by means of the chimney into one or other of the collecting flues F_1 or F_2, according to the position of the reversing apparatus, the amount of air being controlled by dampers D_1 and D_2 or D_3 and D_4 regulated from the inspection galleries. The two cornues supplying the ram side and the flue dampers are shown on the left-hand section of the drawing, the right-hand section showing the evacuation of the burnt gases, according to the principle indicated in the previous diagram. The same firm has also introduced a compound chamber oven possessing all the above features, but designed so as to be capable of being heated by low grade producer gas, blast furnace gas, by its own "rich" gas, or by a mixture of high and low grade gas. The use of individual regenerators and

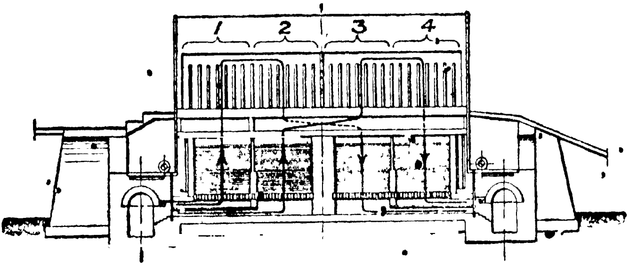


FIG. 72 —Diagram of Latest Coppée System

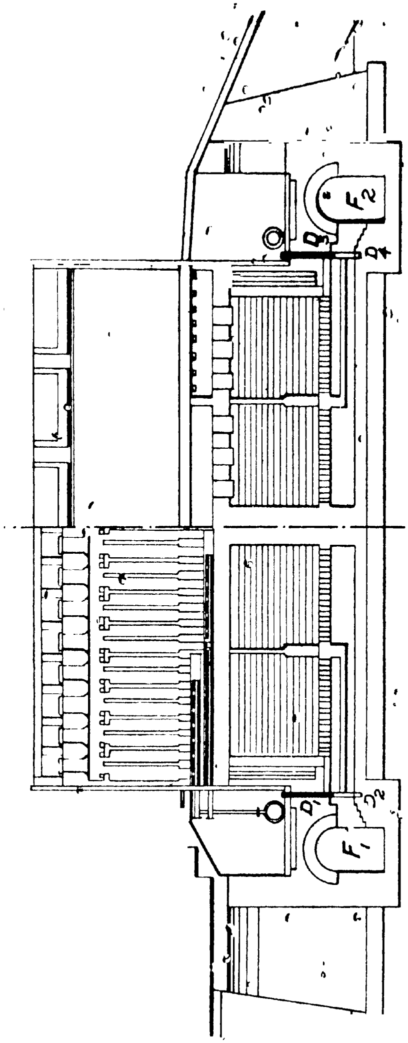


Fig. 73.—Coppée Oven, Individual Regenerator Type

the subdivision of the flues into four sections is adhered to, but in this case the alternative regenerators used for producer gas are divided by a baffle wall, as shown in Fig 74. If rich gas is used to reinforce the temperatures, the cornues C are brought into play. The regenerators K are fed alternately by producer gas, etc., at G and by air at A, the

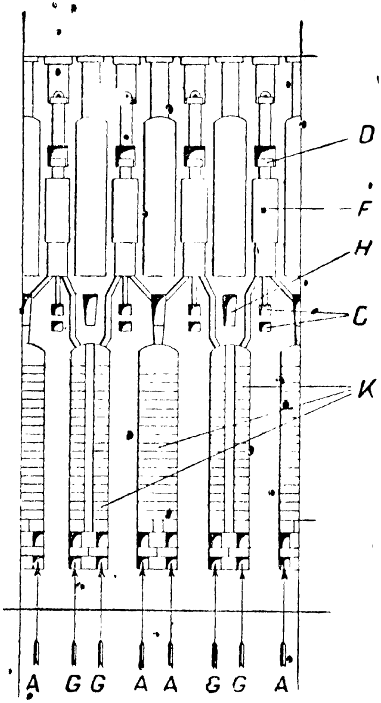
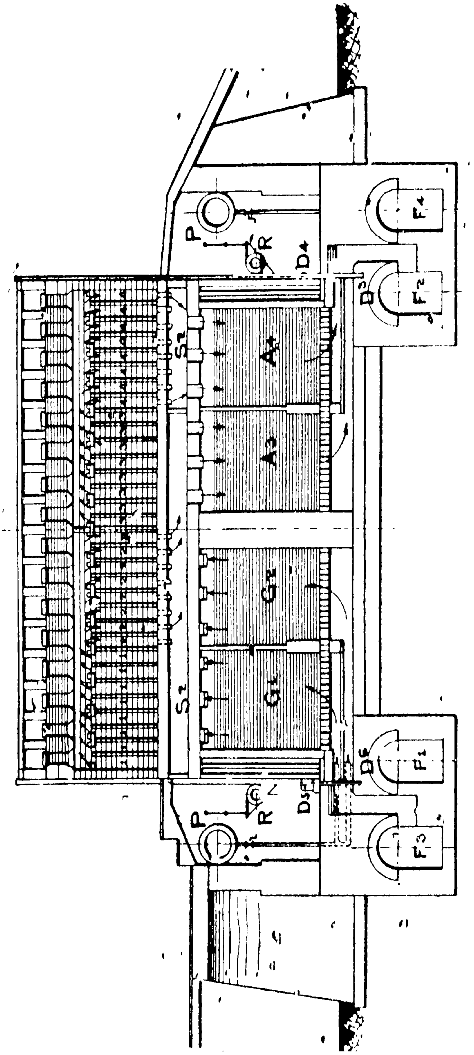
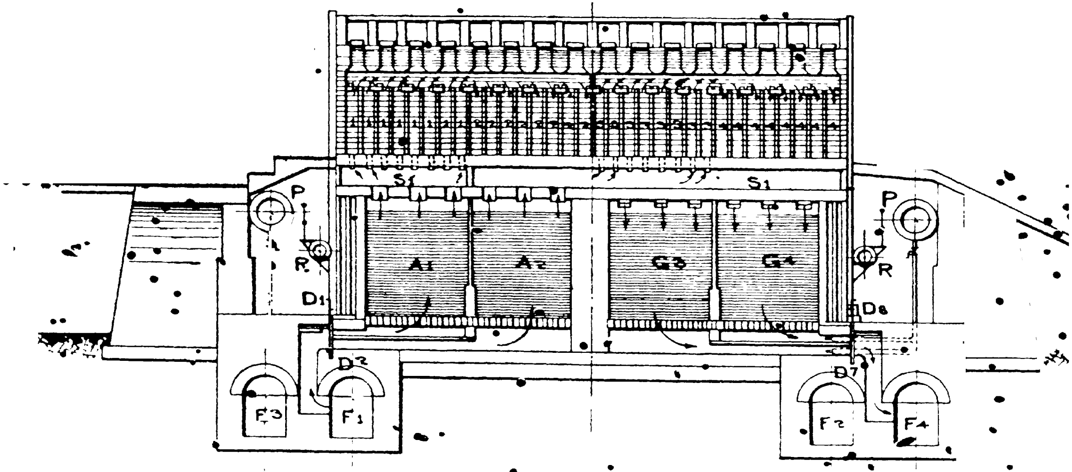


FIG. 74. Coppée Oven Section.

diagram showing the admission of preheated gas and air at the base of the flues.

Figs. 75 and 76 show the general design of this compound oven. Considering these two diagrams in conjunction, it will be seen that producer gas is being heated in G_1 and air in A_1, the two meeting, and combustion ensuing in flues 1, the products of combustion descending in flues 2 and passing through the regenerators A_3 and G_3 to the chimney. Similarly gas and air heated in G_2 and A_2 respectively meet in flues 3,


P
R
D4
F4
D3
F2
S2
A4
A3
G2
G1
S2
D6
F1
F3
D5
R
P



76.—Coppee Oven. General Arrangement of Individual Regenerator.

the burnt gases descending through flues 4 to regenerators G_4 and A_4, passing thence to the chimney. The reversal of these currents is effected by reversing valves at the chimney end of the battery. With the compound oven two sets of main distributing flues are used. The poor gas main is shown at P, the rich gas, when desired being supplied from the smaller main R. By a slight adjustment in the details the flues can be heated by rich gas alone, the whole of the regenerators then being used to preheat air only.

Of recent years the possibilities of blast furnace or producer gas, as the heating agent for coke oven flues, have led the designers of the Koppers oven to adapt their type, where desired, to meet these conditions. In this case blast furnace gas or producer gas, with calorific values of 100 and 140 B.Th.U. per cubic foot respectively, must be used in greater quantity than obtains with coke oven gas, and it is also necessary to preheat these gases as well as the air for combustion to secure the best effect. The Koppers adaptation is shown in Fig. 77. During the preheating stage the regenerators are fed with producer gas and air alternately at G and A. By reason of partition walls the flow is divided, and in approximately equal proportions; producer gas and air, both highly preheated, meet at the base of the heating flues E. Combustion takes place, and, as in their standard regenerative Koppers oven, the heated products of combustion ascend in one-half of the oven wall, descend in the remaining half, giving up their surplus heat to the brickwork of the regenerators underneath, and passing away to the chimney at a temperature in the region of 250° C. The "combination" oven of the same firm is a further development on somewhat similar lines, but allowing the use of a rich or poor gas at will as the heating agent. Thus the oven can be worked as an ordinary coke oven, using a portion of its own gas, or as a gas oven, using producer gas in the flues, the change being readily made without interfering with the normal working of the oven.

The Bagley rapid regenerative oven (Bagley, Mills, & Co.) is specially designed for rapid coking, the coking period varying from sixteen to twenty hours. For this purpose a special quality of refractory material is supplied to deal with the relatively high temperatures and stresses. The higher temperatures obtaining allow a higher yield of gas per ton of coal, whilst, if desired, producer gas, water gas, or furnace gas may be used to heat the flues, the whole of the oven gas being then available for lighting purposes, etc. Individual regenerators are provided for each oven, the regenerators being of the multiple type. The passages leading to and from the various subsections are carefully proportioned to exactly regulate the air requirements, whilst, in conjunction the gas supplies of the individual flues can be regulated by means of specially designed nozzles. In addition, a graduated system of combustion is employed, a primary supply of preheated air being admitted at the base of the flues, a secondary admission higher up completing the combustion.

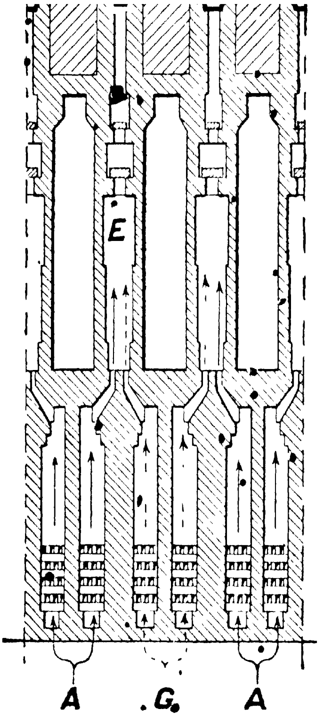


FIG. 77.—Section of Koppers "Gas" Oven.

The general features of this oven are shown in Fig. 78 whilst inset is a diagram showing details of the adjustable nozzle. Heating gas from the main A is delivered to the gas flue B. From thence the gas passes to the superimposed vertical flues by way of the gas nozzles C (shown on a larger scale in the inset). These nozzles are visible and accessible

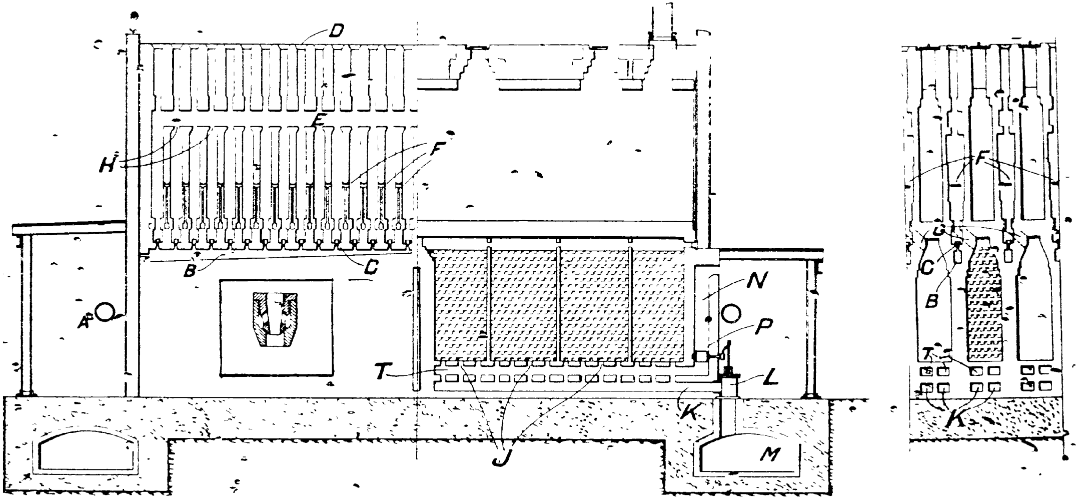


FIG. 78.—B M. RAPID REGENERATIVE OVEN. (Bagley Mills, & Co.)

on removing the caps D, and the flow of gas can be varied through any desired degree by turning the upper and free portion of the nozzle. Preheated air is admitted to the flues at G in volume insufficient for complete combustion, the additional supply requisite for completion being admitted at F. Thus the zone of combustion is lengthened, the tendency for localisation of heat at the base is removed, and more uniform temperature of the oven wall in general is obtained. The openings H at the upper end of the flues are carefully proportioned to ensure equal draught on each flue. The products of combustion pass into the various sections of the regenerators, and by reason of the vertical dividing walls and the relative areas of the outlet passages J, equal volumes of combustion products pass through the respective sections, and thence by way of the canal K and the piston valve L to the waste heat flue M. The flow of gas and air is reversed at intervals in the usual manner. During the intake, the air passes down the communicating flue N, which acts as a heat insulator with corresponding reduction in radiation losses. The air passage is controlled by a wing valve P connected to a common actuating lever, which simultaneously operates the piston valve L

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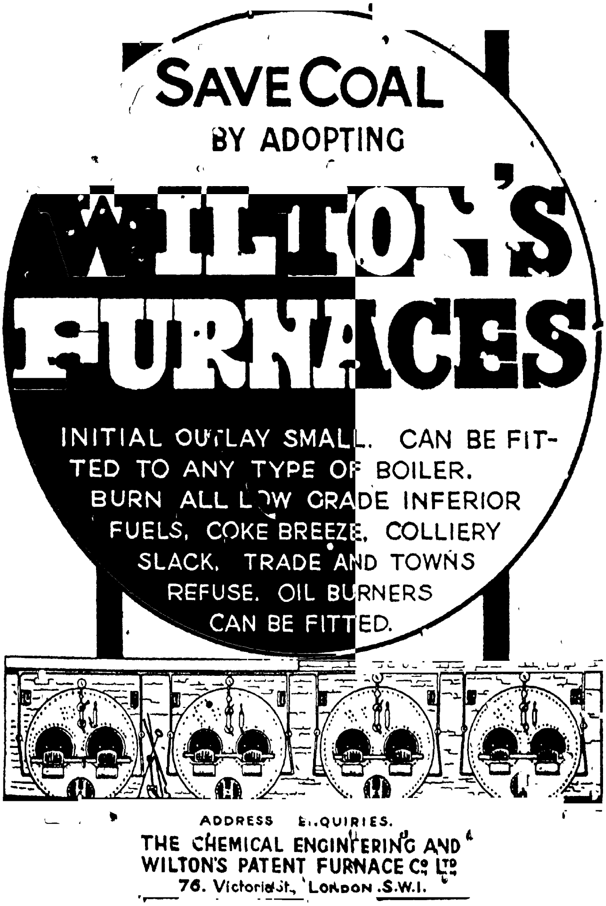

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